REQ. NO. 3-9169

CONTRACT REPORT NO. OSQ83-00123

ENGINEERING SCIENCES DIVISION

Centre for Alternate Fuel Utilization

COMPLETION OF PRE-TRIAL PHASE OF DIESEL EMISSIONS REDUCTION OPTION DEVELOPMENT

Progress Report No. P-4418/1
ES/CAFU/84-15

Submitted to:

Supply & Services Canada
Science Procurement Branch
Place du Portage, Phase III
11 Laurier Street
Hull, Quebec
K1A 0S5

Attention: Dr. R.G. Agarwal

H.C. Vergeer
M. Szabo

March 27, 1984

MICROMEDIA

DEC 19 1984

42AP

TABLE OF CONTENTS

		Page No.
1.	SUMMARY	1
2.	INTRODUCTION	2
3.	SAMPLING SYSTEM DESCRIPTION	3
4.	TEST CELL EQUIPMENT DESCRIPTION	4
5.	ANALYSIS	5
	5.1 PAH's	5
	5.2 Ames Salmonella Mutagenicity Assay	7
6.	TEST RESULTS	7
	6.1 PAH's	8
	6.2 Ames Mutagenic Assay Results	9
	TABLE I - Soluble & Insoluble Particulate Data for PAH Tests	11
	TABLE II - Particulate Loadings and Sample Volumes	12
	TABLE III - Temperature Measurement Summary	13
	TABLE IV - Engine Exhaust Backpressure Data (cm H_2O)	14
	TABLE V - Sample Mass (µg) of PAH's Analyzed in Sampling System	15
	TABLE VI - Method Sensitivity (µg) for PAH Analysis	16
	TABLE VII - Concentration ($\mu g/m^3$) of PAH in Diesel Exhaust	17
	TABLE VIII - PAH Distribution in the Modified Dilution Tube Sampling System	18
	TABLE IX - Corning DPF Collection Efficiency for PAH's	19
	TABLE X - Particulate Distribution - Results for the Ames Test	20
	TABLE XI - Ames Salmonella Mutagenicity Assay Results	21

TABLE OF CONTENTS (cont'd.)

Page No.

FIGURE 1 - Modified Dilution Tube Sampling System Schematic 22

FIGURE 2 - Schematic of Engine Set-Up and Identification of Thermocouple Locations. 23

APPENDIX "A" - HPLC Chromatograms 24

Progress Report No. P-4418/1
ES/CAFU/84-15

1. SUMMARY

A sampling system for the determination of pass-through PAH's and mutagens with the conventional dilution tube has been developed. The sampling system incorporates two polyurethane foam filters (PUF's) installed downstream of the conventional Hi-Vol sampling filter. Effectiveness of the PUF's in collecting the pass-through PAH's was determined with the use of a separate sampling system incorporating an XAD-2 adsorbent cartridge. Three separate test conditions of process blank (background air), neat diesel (without Corning DPF's), and neat diesel with Corning DPF's were used to evaluate the effectiveness of the modified dilution tube.

The Hi-Vol filter collected between 84% to 99% of the individual PAH compounds in the uncontrolled diesel exhaust emissions. With the Corning DPF's installed on the engine the percentage of PAH's collected on the Hi-Vol filter ranged between 37.6% and 92.4% with an average of 78%. Fluoranthene exhibited the greatest tendency to pass-through the Hi-Vol filter. The Corning DPF's reduced the raw exhaust PAH emissions by 90% or better at the steady state engine condition (2200 RPM 7/8 LOAD).

The mutagenic activity of the uncontrolled diesel emissions was 27,338 revertants per cubic meter of raw exhaust with 94% of the mutagenic activity found on the Hi-Vol filter. With the Corning DPF's installed the mutagenic activity of the diesel emissions dropped to 3,660 revertants per cubic meter of raw exhaust resulting in a total mutagenic reduction of 87%. A 5.6% pass-through of mutagens was observed on the PUF's.

2. INTRODUCTION

The sampling and characterization of diesel exhaust emissions has been under constant review and is the subject of optimization studies. Particulates, emitted in the exhaust of diesel engines, are comprised of insoluble and soluble fractions, the latter containing polynucleararomatic hydrocarbon (PAH) compounds. Some of these PAH compounds are known carcinogens and are, therefore, of concern.

Significant advances have been made in the control of diesel exhaust particulate emissions. Exhaust after treatment devices such as ceramic diesel particulate filters (DPF's) are known to trap particulates with an efficiency of around 90%. Although the advances in particulate control technology have been generally laboratory derived, their commercial vehicle application is near. The majority of emissions characterization with the use of DPF's have been carried out under steady state or transient engine operating conditions with little work on the characterization of the products of regeneration. Regeneration is a term describing the oxidation of particulate material accumulated on DPF's or other types of particulate traps.

Dilution tunnels and dilution tubes have generally been used as equipment for the sampling of diesel exhaust particulates. Dilution tunnels accept the complete flow of engine exhaust and a portion of the diluted exhaust is passed through a collection filter. Filters normally used are teflon coated glass fibre. With the dilution tube, a small portion of the exhaust flow is diluted and the complete diluted exhaust flow is passed through a 20 cm by 25 cm high volume sampler (Hi-Vol) filter.

These methods are adequate for collecting particulate emissions from uncontrolled diesel exhaust (i.e. without the use of diesel particulate filters). In the case of particulate controlled diesel exhaust emissions,

however, the reduction of insoluble particulates minimizes the condensation of soluble particulate on the insoluble. It has been theorized, therefore, that the soluble fraction stays in a gaseous or vapour form and passes through the sampling filter. The "pass-through" components may also contain PAH's.

This report summarizes the activities that were carried out to quantitatively determine the pass-through of certain PAH's with the use of a modified dilution tube. The "pass-through" fraction was also analyzed to determine its contribution to results from the Ames mutagenic assay. Tests were carried out to determine background or ambient (also dilution) air conditions prior to engine testing. Baseline engine testing was performed on a Deutz F6L 714 diesel engine operating on No. 1 diesel fuel without the use of diesel particulate filters. Further tests were performed with Corning DPF's installed on the same test engine with the same fuel.

3. SAMPLING SYSTEM DESCRIPTION

The dilution tube, developed under a previous CANMET program, was modified for the determination of "pass-through" PAH's. A section between the high volume filter holder and blower was designed and fabricated. This section was used to hold the polyurethane foam filters (PUF's) downstream of the main sampling filter. Figure 1 gives a schematic of the modified dilution tube sampling system.

A portion of the dilute sample gas is withdrawn downstream of the PUF's and passed through an XAD-2 resin trap. Sample gas flow was regulated using a rotameter and measured using a calibrated, temperature compensated dry gas meter. The total volume sampled with the XAD-2 sampling system was approximately 1% to 1.8% of the total volume sampled with the modified dilution tube. The XAD-2 sampling system was not used in the collection of samples for the Ames mutagenic assay.

Diesel exhaust gases were diluted with air (dyno cell control room air) at an approximate ratio of 16:1. Dilution ratios were determined by measuring the raw exhaust gas carbon dioxide (CO_2) concentration and the dilute CO_2 concentration in the dilution tube. A control valve in the raw exhaust transfer tube was used to regulate the flow of exhaust gas while a total dilute exhaust flow was controlled by adjusting the Hi-Vol blower speed.

Each test constituted the collection of dilute exhaust particulate on a total of eight Hi-Vol filters. Sampling duration for each filter was between 21 to 30 minutes depending on the pressure drop across the sampling filter.

4. TEST CELL EQUIPMENT DESCRIPTION

All tests were conducted using an air-cooled Deutz F6L 714 diesel engine. A Go-Power, DT-1000, flow through water brake dynamometer was used to apply load to the test engine. This dynamometer is attached directly to the flywheel housing. Two oil bath type air cleaners were used in the combustion air stream. Fifteen type 'K' thermocouples were used to measure temperatures at various points on the test engine as shown in Figure 2. Direct temperature readings were taken off a Doric digital trendicator with thermocouple switching capability.

The test engine was operated at 2200 RPM and 7/8 load (373 N.m torque) for each test. No. 1 grade diesel fuel was used for all tests.

Corning ceramic diesel particulate filters were used for the controlled diesel emissions tests. The DPF's, measuring 30 cm in diameter by 30 cm long, were designed for a Deutz F8L 413F/W diesel engine. It was assumed, however, that these slightly oversized DPF's would represent similar operating characteristics to a properly sized unit at steady state engine operating conditions. DPF's were preconditioned prior to each test by operating the engine at full load/speed condition. This preconditioning procedure ensured that full regeneration of the DPF's had taken place.

Engine exhaust backpressures, upstream and downstream of the DPF's, were measured during each test. In all cases, differential exhaust backpressure was not allowed to exceed 15 cm of water.

5. ANALYSIS

5.1 PAH's

XAD-2 resin used for the PAH tests was cleaned prior to sampling. The resin was cleaned by extraction in a Soxhlet apparatus for 20 hours using spectroscopic grade cyclohexane. A rotary evaporator was then used to evaporate the cyclohexane extract to dryness under reduced pressure at a temperature of 30°C. The dryed residue was then dissolved in 5 to 10 mL of methanol, and a sample injected into a high performance liquid chromatograph (HPLC) for the quantitative determination of the following PAH compounds:

- fluoranthene (F)
- benzo[a]anthracene (B[a]A)
- benzo[k]fluoranthene (B[k]F)
- benzo[a]pyrene (B[a]P)
- dibenz[ah]anthracene (DiBenz[ah]A)

and
- benz[ghi]perylene (Benz[ghi]Per).

The following HPLC conditions were used for the determination of the PAH compounds:

- Injection Volume : 50 µL
- Column : C_{18} RP Ultrasphere ODS
- 4.6 mm x 25 cm
- Gradient Program : 50% CH_3CN -100% CH_3CN in 50 min. Hold at 100% CH_3 for 15 min. 100% CH_3CN 50% CH_3CN in 10 min.
- Flow Rate : 1.00 mL/min

• Detector	:	Fluorescence, λex 280 nm λam 389 nm. Sensitivity 5.61 Attenuation 0.5 μA.
• Pressure	:	190-1850 psi
• Data System	:	SP-4100 computing Integrator
• Chart Speed	:	0.5 cm/min
• Temperature	:	Ambient

Samples of extracts from the cleaning of the XAD-2 resin were analyzed to ensure that impurities did not interfere with sample analysis. HPLC chromatograms for the process blank and test samples are presented in Appendix A.

Polyurethane foam filters were also cleaned prior to sampling. Several extractions of the PUF's with cyclohexane were necessary prior to obtaining clean filters.

Following sampling, the XAD-2 resin and glass wool support were removed from their container and extracted for 8 hours. Samples were extracted with cyclohexane in a Soxhlet apparatus. The PUF plugs were cut into small sections and extracted in a Soxhlet apparatus for 8 hours with cyclohexane. Hi-vol filters were extracted for 12 hours in dichloromethane in a Soxhlet apparatus.

Individual cyclohexane and dichloromethane extracts were evaporated to dryness on a rotary evaporator under reduced pressure at a temperature of 35°C. The resultant residue was dissolved in 2 mL of methanol and a sample of the methanol solution injected into the HPLC.

5.2 Ames Salmonella Mutagenicity Assay

Three sets of Hi-Vol filters and polyurathane foam plugs were assayed for mutagenicity using the Ames Salmonella mutagenicity assay. The Salmonella tester strain used was TA-98 in the absence of metabolic activation. The three samples were 1) Blank Dilution Air, 2) Raw Diesel Exhaust and 3) Diesel plus the Corning Filter Traps. The Hi-Vol filters and PUF (polyurathane foam plugs) were extracted for 16 hr with dichloromethane (DCM). The solvent was then taken to dryness and the weight of the soluble organic fraction (SOF) determined. The samples were then dissolved in dimethyl sulfide (DMSO) at a concentration of 200 µg/mL and submitted for testing in the Ames Salmonella mutagenicity assay. Testing was carried out between 0 and 1000 µg per plate to obtain dose-response data. The specific activity as revertants/µg was calculated for each of the samples from the linear portion of the dose response curve.

6. TEST RESULTS

Selected Hi-Vol filters were analyzed to determine the soluble and insoluble fractions of the total particulate material. For tests carried out on uncontrolled engine emissions the soluble fraction accounted for approximately 44% of the total particulate (see Table 1). With the Corning DPF's installed the soluble fraction averaged 65% of the total particulate. Table 2 gives the volumes sampled for the XAD-2 and dilution tube sampling systems. Particulate loadings for the three test conditions are also presented in Table 2. Based on neat diesel and neat diesel with DPF tests the total particulate control efficiencies averaged 80% and 93%.

Table 3 gives the average temperature measurements for each test condition. Thermocouple identifications are given in Figure 2. Table 4 gives the engine exhaust backpressure data recorded for each sample filter (total 8 sample filters) for the three test conditions.

6.1 PAH's

The XAD-2 cartridge, PUF plugs, and Hi-Vol filters were analyzed for the 6 PAH's for the process blank, neat diesel, and neat diesel with Corning DPF's. Table 5 gives the sample mass of PAH's analyzed at the three points in the modified dilution tube sampling system. The HPLC method sensitivities (lower detection limits) for the 6 PAH compounds are given in Table 6. Variations in the method sensitivity for a given PAH compound are due to variations in the sample dilutions.

Concentrations of PAH's in the raw diesel exhaust (corrected for dilution ratio) are presented in Table 7. Minor quantities of 5 of the 6 PAH compounds were found in the process blank (i.e. no diesel exhaust entering the dilution tube). These data suggest that the dilution air does not contribute a sampling artifact to the results found with diesel exhaust entering the dilution tube.

In the case of uncontrolled diesel exhaust emissions the majority of the PAH compounds were found on the Hi-Vol sampling filter. The Hi-Vol sampling filters collected between 84.4% and 99.3% of the total individual PAH compounds measured at the three locations in the dilution tube. Polyurethane foam filters collected 9.7% of the pass-through fluoranthene and 13.1% of pass-through benzo[a]anthracene. Minimal quantities of benzo[K]fluoranthene and benzo[a]pyrene (i.e. < 2.7%) were found on the PUF's. Also, minimal quantities of PAH compounds were found on the XAD-2 resin traps. These data show that the polyurethane foam filters are reasonably efficient in collecting the minimal quantities of PAH's that pass-through the Hi-Vol sampling filter.

Concentrations ($\mu g/m^3$) of PAH's are significantly lower when the Corning DPF's are installed on the Deutz F6L 714 engine exhaust. The most significant pass-through was observed for fluoranthene where 49% was found in the XAD-2

resin. This result is somewhat similar to that found in the process blank (52%). It is assumed, therefore, that fluoranthene exhibits a high propensity to adsorb onto diesel particulate matter. The polyurethane filters were efficient in collecting (1% to 18%) of the 5 other PAH compounds that passed through the Hi-Vol sampling filter.

Table 8 shows that there is a slightly higher pass-through of PAH's for test conducted with the Corning DPF's. Although the percentage pass-through is greater, the quantities of PAH's found in all three locations are significantly lower than for tests without the Corning DPF's. PAH collection efficiencies were determined at 90% or better.

6.2 Ames Mutagenic Assay Results

Table 10 gives results of the particulate distribution for the three test conditions of process blank, neat diesel, and neat diesel with Corning DPF's. Total particulate concentration was 89.3 mg/m^3 for neat diesel as measured on the Hi-Vol filter. A pass-through of 10.9 mg/m^3 was found on the PUF's. Soluble organic fraction accounted for 39.9% of the total particulate on the Hi-Vol filter and 76.2% on the PUF's. The Hi-Vol filter collected 81.2% of the total soluble material with 18.8% collected on the PUF's.

The total particulate concentration was 6.2 mg/m^3 as determined with the Hi-Vol filter for engine operation with the Corning DPF's installed. A further 5.3 mg/m^3 of total particulate material was found on the PUF's under the same test conditions. The soluble particulate fraction accounted for 60.0% of the total particulate material on the Hi-Vol filter and 64.0% on the PUF's. Based on these data, the total particulate collection efficiency for the Corning DPF's is 89% and 84% for soluble particulate

Ames Salmonalla mutagenicity assay results are presented in Table 2. The process blank produced a specific activity of 0.073 revertants per microgram

of soluble particulate on the Hi-Vol filter. A negative mutagenic response was determined for the soluble fraction collected on the PUF's. The mutagenic contribution of the process blank to diesel emission test results is negligible.

A specific mutagenic activity of 0.72 revertants per microgram of soluble particulate was found on the Hi-Vol filter from the neat diesel tests (i.e. without Corning DPF's). This revertant concentration results in a concentration of 25,704 revertants per cubic meter of raw exhaust. The specific activity of the soluble fraction collected with the PUF's was significantly lower at 0.19 resulting in 1,634 revertants per cubic meter of raw diesel exhaust. Therefore, six percent of the total mutagenic activity was attributable to pass-through mutagens.

In the case of tests carried out with the Corning DPF's installed, the specific activity (0.96) was higher than the neat diesel test. However, since the soluble concentration is lower, the revertants per cubic meter of exhaust is only 3,456. The specific activity on the PUF's was very low at 0.06 revertants per microgram of soluble particulate. The total pass-through mutagens accounted for 5.6% of the total mutagenicity. An overall reduction in the total mutagenicity of 87% can be attributed to the effective control efficiency of the Corning DPF's

M. Szabo
Technologist I
Centre for Alternate Fuel Utilization
Engineering Sciences Division

H.C. Vergeer
Senior Technologist
Centre for Alternate Fuel Utilization
Engineering Sciences Division

:sw

TABLE I

Soluble & Insoluble
Particulate Data for PAH Tests

Test 2, Neat Diesel Filter #	Condition RPM/lb.ft.	Insoluble Fraction (%)	Soluble Fraction (%)	Total Weight (mg)	PUF plugs Wt (mg)
1	2200/275	57.4	42.6	90.3	41.0
2	2200/275	60.1	39.9	87.4	
3	2200/275	54.1	45.9	84.4	
4	2200/275	54.2	45.8	80.2	
5	2200/275	57.9	42.1	83.8	
6	2200/275	55.7	44.3	75.7	
7	2200/275	45.3	54.7	79.0	
8	2200/275	61.3	38.7	86.9	
Test 3, Neat Diesel with Corning DPF's					
1	2200/275	34.9	65.1	12.9	117.0
2	2200/275	49.0	51.0	14.7	
3	2200/275	47.1	52.9	14.0	
4	2200/275	53.8	46.2	13.2	
5	2200/275	26.3	73.7	9.9	
6	2200/275	39.6	60.4	9.1	
7	2200/275	20.0	80.0	8.0	
8	2200/275	10.0	90.0	8.0	

TABLE II

Particulate Loadings
and
Sample Volumes

	Process Blank		Neat Diesel		Neat Diesel with DPF's	
	XAD-2	PUF & Filter	XAD-2	PUF & Filter	XAD-2	PUF & Filter
Test 1 (PAH)	3.363 m^3	188.41 m^3				
Loading (mg/m^3)		0.261 (.116)*				
Test 2 (PAH)			2.228 m^3	143.85 m^3		
Loading (mg/m^3)				85.23 (80.30)		
Test 3 (PAH)					2.014 m^3	207.69 m^3
Loading (mg/m^3)						17.38 (7.13)
Test 1 (AMES)	-	210.04 m^3				
Loading (mg/m^3)		0.188 (.136)				
Test 2 (AMES)			-	146.53 m^3		
Loading (mg/m^3)				100.12 (89.26)		
Test 3 (AMES)					-	207.67 m^3
Loading (mg/m^3)						11.50 (6.18)

* Loading Based on Particulate Collected on Hi-Vol Filters Only.

TABLE III

Temperature Measurement Summary

Thermocouple #	PAH TESTS			AMES TESTS		
	Test 1 Process Blank (°C)	Test 2 Neat Diesel (°C)	Test 3 Neat Diesel With DPF's (°C)	Test 1 Process Blank (°C)	Test 2 Neat Diesel (°C)	Test 3 Neat Diesel With DPF's (°C)
1	425	452	423	423	428	427
2	449	477	452	450	452	461
3	444	474	448	448	446	454
4	432	453	440	429	432	444
5	427	-	-	-	-	-
6	442	464	444	439	445	446
7	445	474	415	446	448	422
8	458	483	417	454	462	420
9	21	29	24	15	25	25
10	49 (Room Temp)	56 (Room Temp)	417	57 (Room Temp)	50 (Room Temp)	423
11	45 (Room Temp)	51 (Room Temp)	402	52 (Room Temp)	45 (Room Temp)	404
12	28	46	45	31	43	44
13	25	28	27	29	25	27
14	194	207	205	196	224	210
15	90	96	94	94	90	95

*Use legend page for thermocouple numbers

TABLE IV

Engine Exhaust Backpressure Data (cm H_2O)

Test No.	Location	Sampling Filter Number 1	2	3	4	5	6	7	8
		PAH TESTS							
1. Process Blank	Left Exhaust	14.2	14.2	14.6	14.6	14.2	15.2	15.8	14.0
1. Process Blank	Right Exhaust	9.1	8.8	8.4	8.8	9.0	9.6	11.0	9.8
2. Neat Diesel	Left Exhaust	11.4	11.6	13.2	13.4	13.4	12.6	15.8	16.6
2. Neat Diesel	Right Exhaust	9.8	9.6	9.8	10.0	10.2	10.0	10.4	11.2
3. Neat Diesel w DPF's	Before Left DPF	21.7	22.5	23.6	24.0	24.7	25.6	26.4	27.6
3. " " " " " "	After Left DPF	9.4	9.4	9.4	9.4	9.4	9.2	9.3	9.2
3. " " " " " "	Before Right DPF	25.0	26.6	28.2	29.5	30.9	31.8	32.7	34.2
3. " " " " " "	After Right DPF	8.1	9.9	12.4	10.6	9.2	6.0	7.9	9.4
		AMES TESTS							
1. Process Blank	Left Exhaust	18.2	18.2	18.2	19.0	18.2	18.2	17.8	15.0
1. Process Blank	Right Echaust	10.6	11.0	11.2	11.8	12.0	11.2	11.2	9.9
2. Neat Diesel	Left Exhaust	13.2	13.8	13.8	14.4	14.2	13.0	13.2	14.6
2. Neat Diesel	Right Exhaust	10.2	10.0	10.2	10.4	10.4	9.6	10.2	10.2
3. Neat Diesel w DPF's	Before Left DPF	25.2	27.8	29.2	31.0	23.4	25.0	27.0	29.1
3. " " " " " "	After Left DPF	9.0	9.0	9.0	9.2	9.0	9.0	9.0	9.1
3. " " " " " "	Before Right DPF	32.5	35.4	37.6	40.6	32.2	34.8	36.6	39.0
3. " " " " " "	After Right DPF	9.2	6.6	8.6	11.4	7.1	12.6	8.8	12.2

TABLE V

Sample Mass (μg) of PAH's Analyzed in Sampling System

PAH	Process Blank			Neat Diesel			Neat Diesel with DPF's		
	XAD-2 Cartridge	PUF Plugs	Filter	XAD-2 Cartridge	PUF Plugs	Filter	XAD-2 Cartridge	PUF Plugs	Filter
F	0.0296	1.289	0.227	0.0206	4.541	41.053	0.0299	0.8366	2.360
B[a]A	0.0086	0.343	0.100	0.0136	4.677	30.020	ND	4.7902	4.262
B[k]F	0.0014	0.0030	0.084	ND	0.3885	15.455	ND	0.2198	2.041
B[a]P	0.0036	0.0979	0.082	ND	1.5726	58.294	ND	0.2248	6.273
Dibenz[ah]A	ND*	ND	ND	ND	ND	9.041	ND	ND	ND
Benz[ghi]Per	ND	0.1081	0.123	ND	0.4365	118.050	ND	ND	14.458

* None detected

TABLE VI

Method Sensitivity (μg) for PAH Analysis

PAH	Process Blank			Neat Diesel			Neat Diesel with DPF's		
	XAD-2 Cartridge	PUF Plugs	Filter	XAD-2 Cartridge	PUF Plugs	Filter	XAD-2 Cartridge	PUF Plugs	Filter
F	0.0024	0.0241	0.032	0.0024	0.0482	1.000	0.0024	0.0482	0.080
B[a]A	0.0020	0.0198	0.024	0.0020	0.0396	0.750	0.0020	0.0396	0.060
B[k]F	0.0013	0.0135	0.014	0.0013	0.0270	0.437	0.0013	0.0270	0.035
B[a]P	0.0030	0.0518	0.062	0.0030	0.1036	1.937	0.0030	0.1036	0.155
Dibenz[ah]A	0.0033	0.0330	0.038	0.0033	0.0660	1.187	0.0033	0.0660	0.095
Benz[ghi]Per	0.0097	0.0967	0.116	0.0097	0.1934	3.625	0.0097	0.1934	0.290

TABLE VII

Concentration ($\mu g/m^3$) of PAH in Diesel Exhaust

PAH	Process Blank			Neat Diesel			Neat Diesel with DPF's		
	XAD-2 Cartridge	PUF Plugs	Filter	XAD-2 Cartridge	PUF Plugs	Filter	XAD-2 Cartridge	PUF Plugs	Filter
F	0.0088	0.0068	0.0012	0.1600	0.5461	4.9372	0.2449	0.0665	0.1875
B[a]A	0.0026	0.00182	0.00053	0.1056	0.5625	3.6103	<0.0164	0.0794	0.3386
B[k]F	0.00042	0.000016	0.00044	<0.0101	0.0467	1.8587	<0.0011	0.0175	0.1621
B[a]P	0.0011	0.00052	0.00044	<0.0233	0.1891	7.0107	<0.0246	0.0178	0.4984
Dibenz[ah]A	<0.00098*	<0.00018	<0.00020	<0.0256	<0.0079	1.0873	<0.0270	<0.0052	<0.0075
Benz[ghi]Per	<0.00288	0.00057	0.00065	<0.0753	<0.0232	14.1972	<0.0794	<0.0154	1.1486

* These values obtained using the lower detection limit and dilution ratio.

TABLE VIII

PAH Distribution in the Modified Dilution Tube Sampling System

PAH	Process Blank			Neat Diesel			Neat Diesel with DPF's		
	XAD-2 Cartridge	PUF Plugs	Filter	XAD-2 Cartridge	PUF Plugs	Filter	XAD-2 Cartridge	PUF Plugs	Filter
F	52.4%	40.5%	7.1%	2.8%	9.7%	87.5%	49.1%	13.3%	37.6%
B[a]A	52.5%	36.8%	10.7%	2.5%	13.1%	84.4%	3.8%*	18.3%	77.9%
B[k]F	48.0%	1.8%	50.2%	0.5%*	2.4%	97.0%	0.6%*	9.7%	89.7%
B[a]P	53.4%	25.2%	21.4%	0.3%*	2.6%	97.1%	4.5%*	3.3%	92.2%
Dibenz[ah]A	-	-	-	2.3%*	0.7%*	97.0%	-	-	-
Benz[ghi]Per	70.2%*	13.9%	15.9%	0.5%*	0.2%*	99.3%	6.4%*	1.2%*	92.4%

*Percentages obtained using lower detection limit values

TABLE IX

Corning DPF Collection Efficiency for PAH's

PAH COMPOUND	Neat Diesel Total $\mu g/m^3$	Neat Diesel with DPF's Total $\mu g/m^3$	Corning DPF Collection Efficiency For PAH's
Fluoranthene	5.6433	0.4989	91%
Benzo (a) anthrancene	4.2784	0.4344	90%
Benzo (k) fluoranthene	1.9155	0.1807	91%
Benzo (a) pyrene	7.2231	0.5408	93%
Di benz (ah) anthrancene	1.1208	<0.0397	>96%
Benz (ghi) perylene	14.2957	1.2434	91%

TABLE X

Particulate Distribution - Results for the Ames Test

	Process Blank		Neat Diesel		Neat Diesel with Corning DPF's	
	Hi-vol filter	PUF's	Hi-vol filter	PUF's	Hi-vol filters	PUF's
*Total particulate mg/m^3	0.136	0.0520	89.3	10.9	6.2	5.3
Soluble particulate mg/m^3	0.128	0.0657	35.7	8.3	3.6	3.4
Insoluble particulate mg/m^3	0.008	0	53.6	2.6	2.6	1.9
% Soluble of total particulate	93.7	100	39.9	76.2	60.0	64.0
% Soluble collected on hi-vol filters	66.0		81.2		51.3	
% Soluble collected on PUF's		34.0		18.8		48.7
Dilution Ratio	-		16.0:1		16.8:1	

* Concentrations are corrected for dilution ratio to raw exhaust concentrations.

TABLE XI

Ames Salmonella Mutagenicity Assay Results

	Process Blank		Neat Diesel		Neat Diesel with Corning DPF's	
	Hi-vol filter	PUF's	Hi-vol filter	PUF's	Hi-vol filter	PUF's
Specific Activity Revertants/ µg of soluble particulates	0.073	-	0.72	0.19	0.96	0.06
*Revertants/m^3	9.34	-	25704	1634	3456	204
*Total Mutagenicity in Revertants/m^3	9.34		27338		3660	
% Mutagenicity on Hi-vol filters	100%		94.0		94.4	
% Mutagenicity on PUF's		0		6.0		5.6

* Corrected for dilution ratio to raw diesel exhaust concentrations.

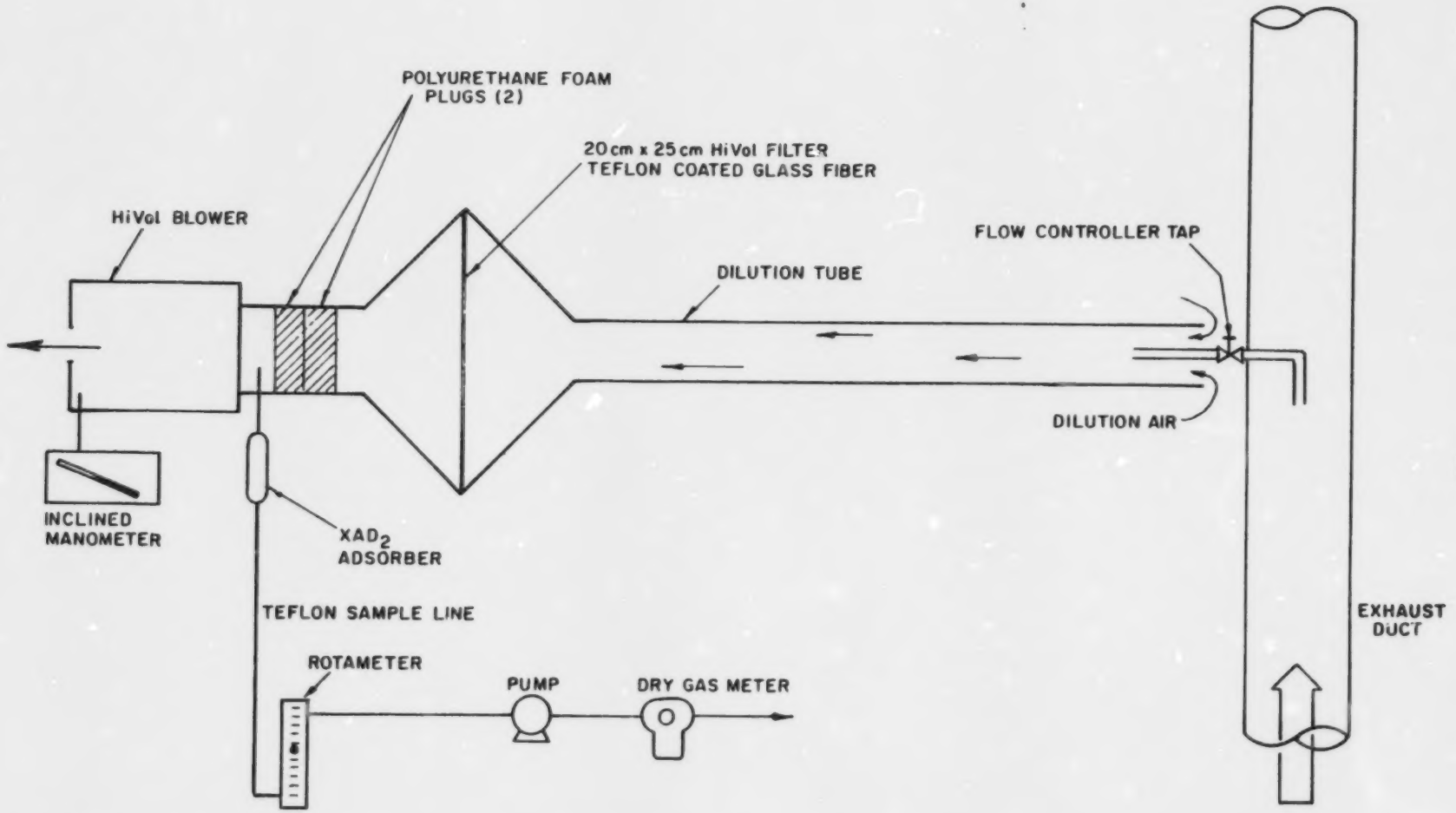
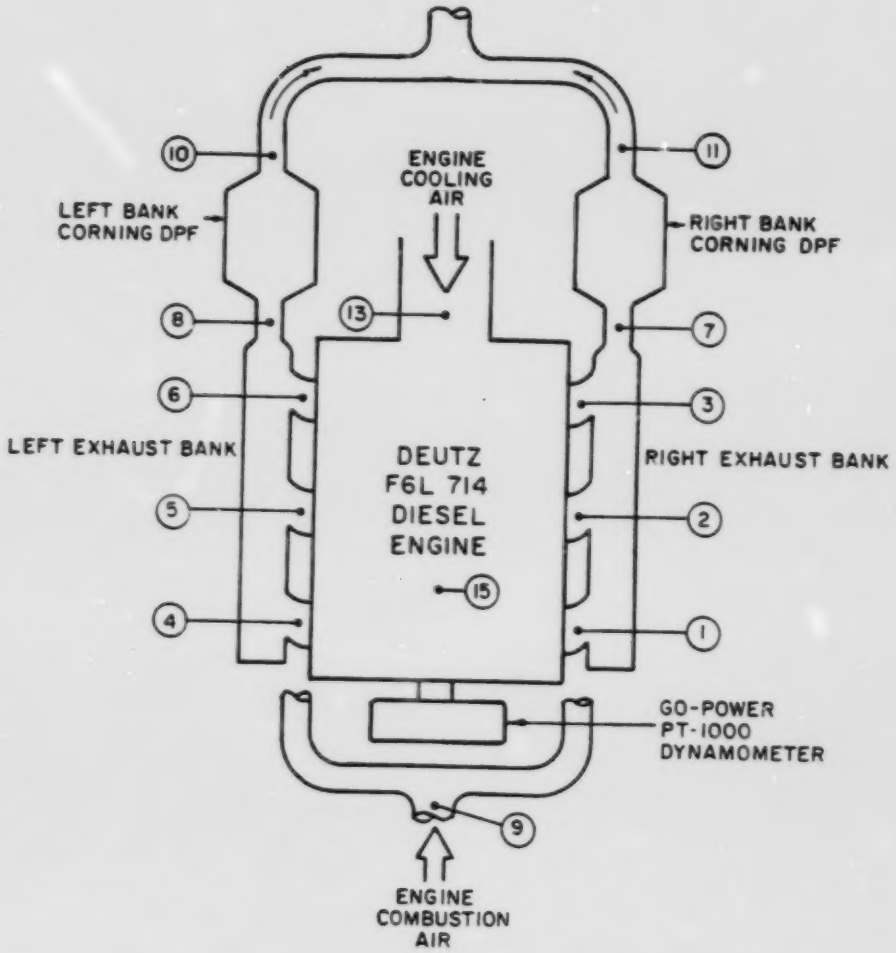


FIG. 1 MODIFIED DILUTION TUBE SAMPLING SYSTEM SCHEMATIC



THERMOCOUPLE No.	LOCATION
1	EXHAUST TEMPERATURE CYLINDER No.
2	" " " "
3	" " " "
4	" " " "
5	" " " "
6	" " " "
7	EXHAUST TEMPERATURE BEFORE LEFT DPF
8	" " " RIGHT DPF
9	COMBUSTION AIR TEMPERATURE
10	EXHAUST TEMPERATURE AFTER LEFT DPF
11	" " " RIGHT DPF
12	DILUTION TUBE TEMPERATURE
13	ENGINE COOLING AIR TEMPERATURE
14	EXHAUST TRANSFER TUBE TEMPERATURE
15	ENGINE CRANKCASE OIL TEMPERATURE

FIG. 2 SCHEMATIC OF ENGINE SETUP AND IDENTIFICATION OF THERMOCOUPLE LOCATIONS.

APPENDIX "A"

HPLC Chromatograms

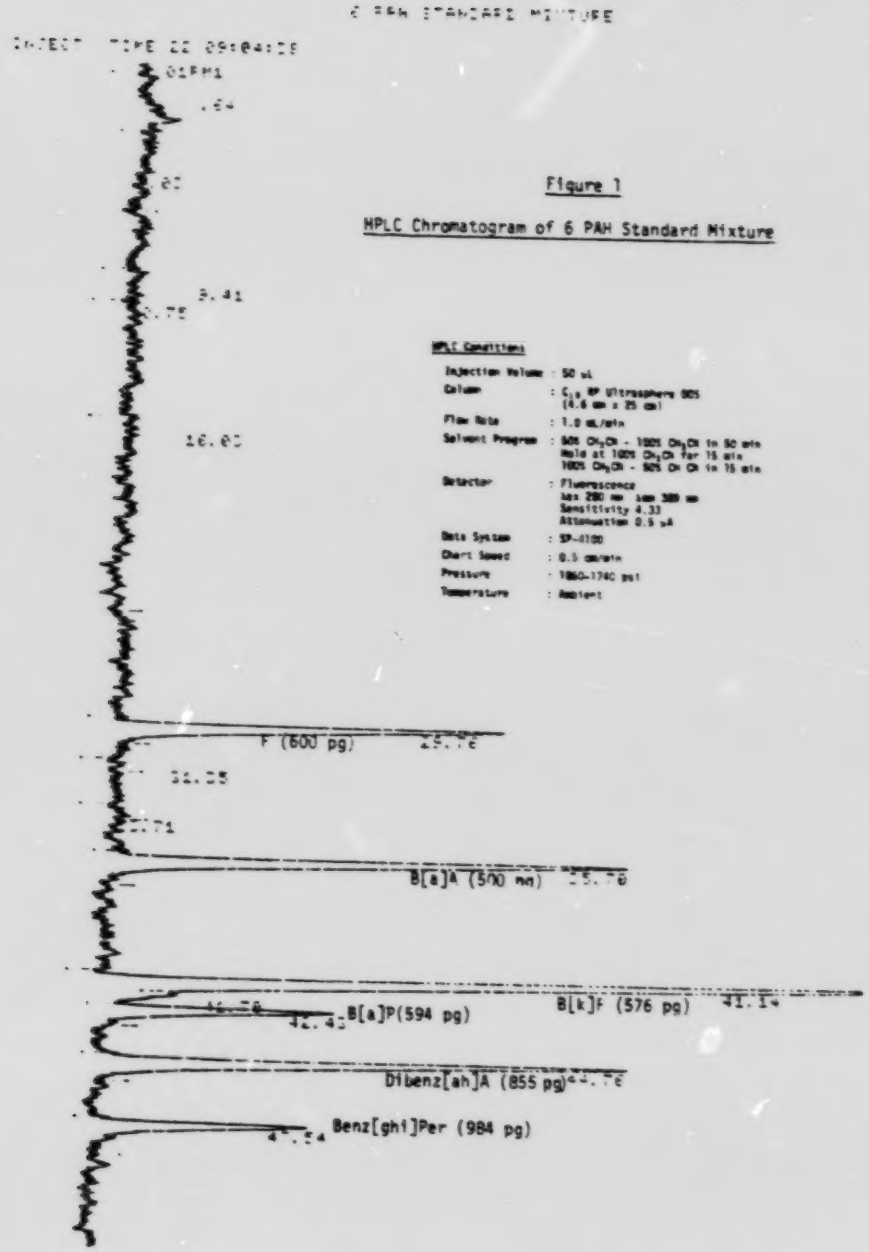


Figure 1

HPLC Chromatogram of 6 PAH Standard Mixture

HPLC Conditions:

Injection Volume	: 50 uL
Column	: C_{18} BP Ultrasphere ODS (4.6 mm x 25 cm)
Flow Rate	: 1.0 mL/min
Solvent Program	: 60% CH_3CN - 100% CH_3CN in 30 min Hold at 100% CH_3CN for 15 min 100% CH_3CN - 60% CH_3CN in 15 min
Detector	: Fluorescence λ_{ex} 280 nm λ_{em} 389 nm Sensitivity 4.33 Attenuation 0.5 uA
Data System	: SP-4100
Chart Speed	: 0.5 cm/min
Pressure	: 1860-1740 psi
Temperature	: Ambient

FIGURE 2

HPLC Chromatogram of PUF Extract - Second Set

(HPLC Conditions same as Fig. 1)

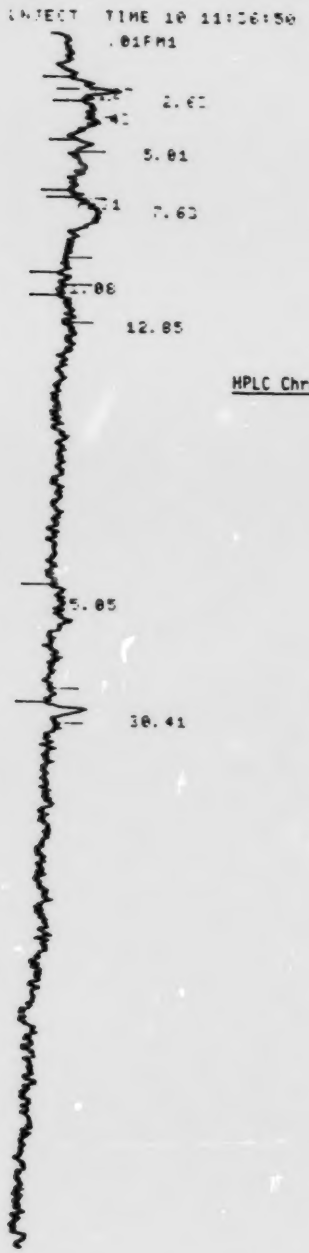


FIGURE 3

HPLC Chromatogram of PUF Extract - Last Extract of Second Set

(HPLC Conditions same as Fig. 1)

FIGURE 4

HPLC Chromatogram of PUF Extract - Third Set

(HPLC Conditions same as Fig.1)

FIGURE 5

HPLC Chromatogram of PUF Extract - Fourth Set

(HPLC Conditions same as Fig. 1)

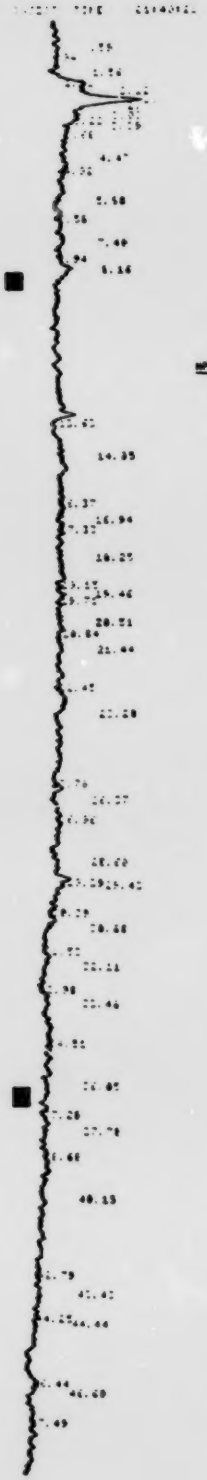


FIGURE 6

HPLC Chromatogram of XAD-2 Resin Extract

(HPLC Conditions same as Fig. 1)

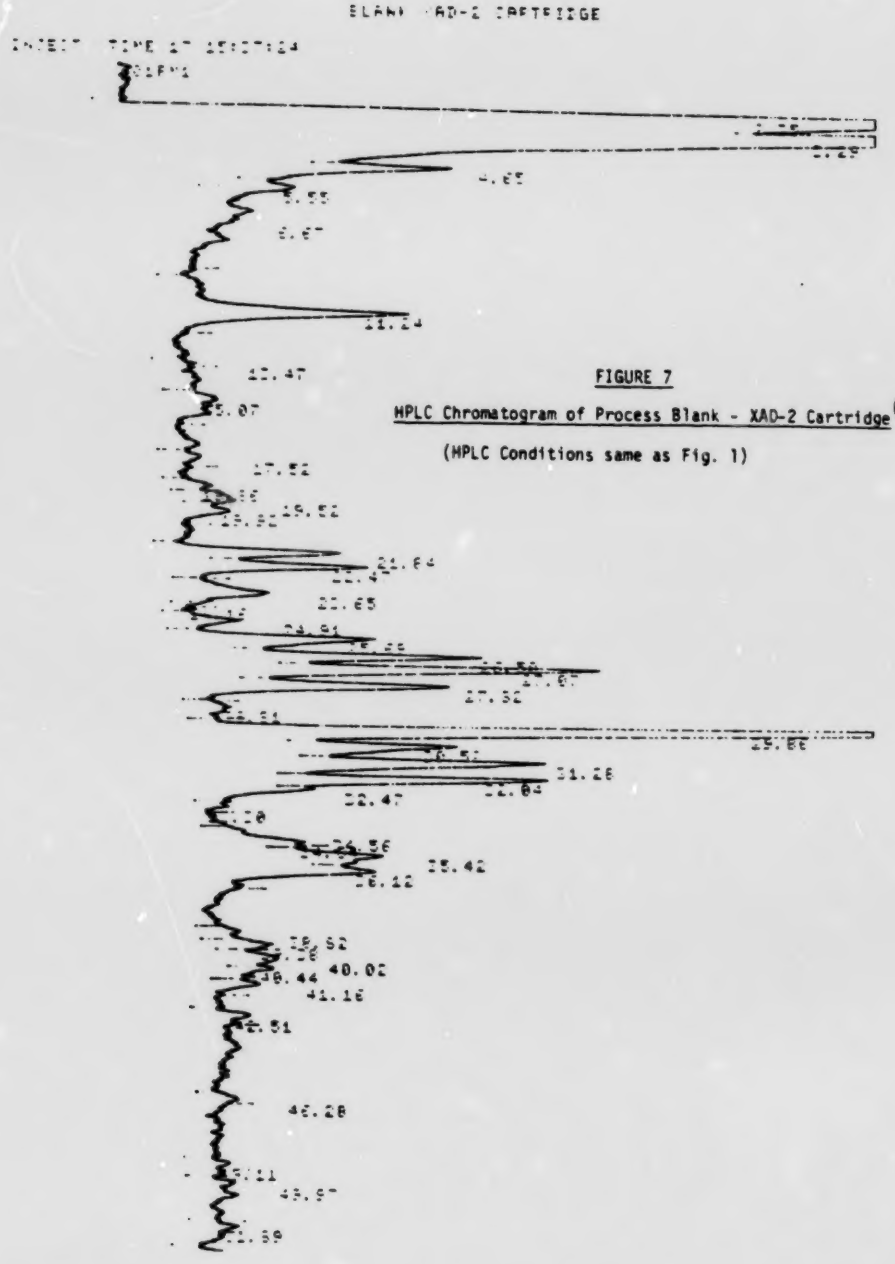


FIGURE 7

HPLC Chromatogram of Process Blank - XAD-2 Cartridge

(HPLC Conditions same as Fig. 1)

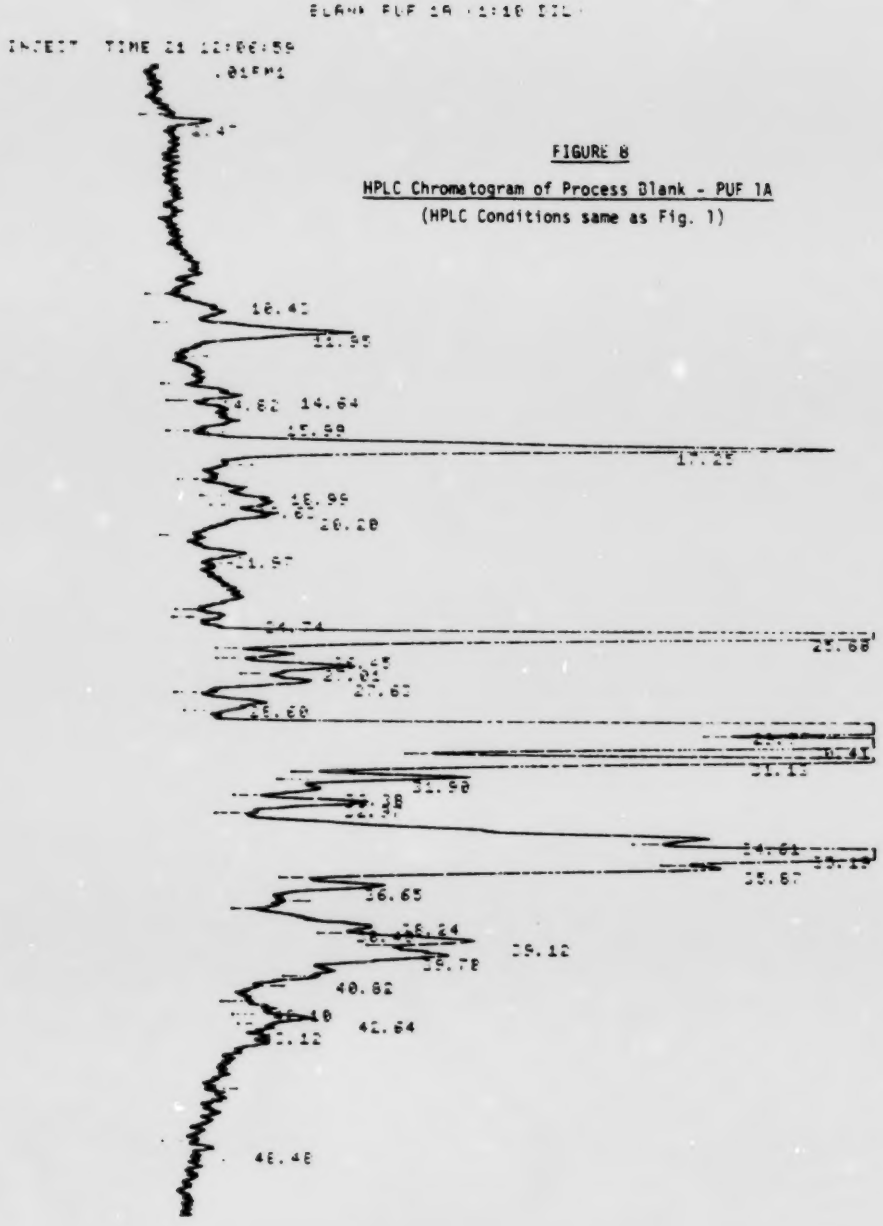


FIGURE 8

HPLC Chromatogram of Process Blank - PUF 1A

(HPLC Conditions same as Fig. 1)

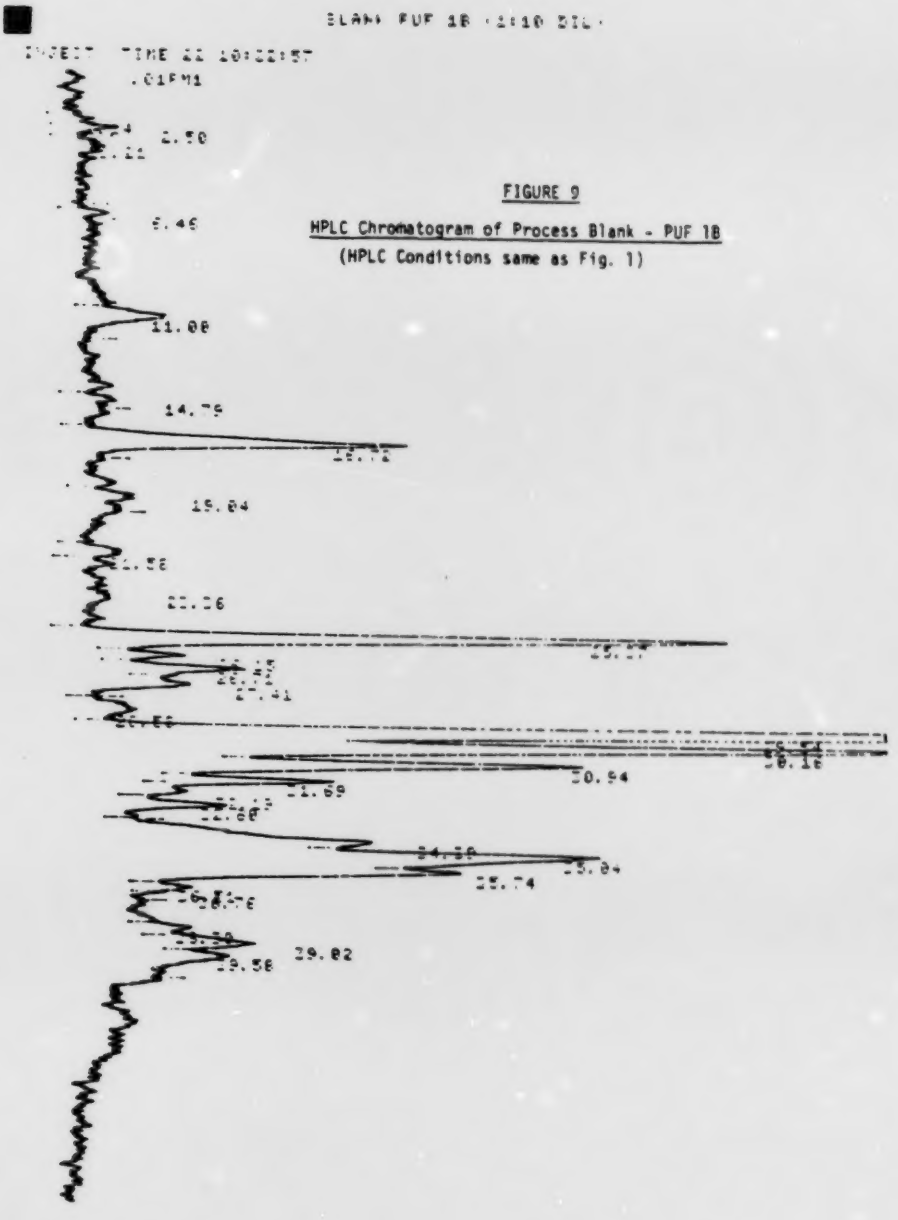


FIGURE 9

HPLC Chromatogram of Process Blank - PUF 1B

(HPLC Conditions same as Fig. 1)

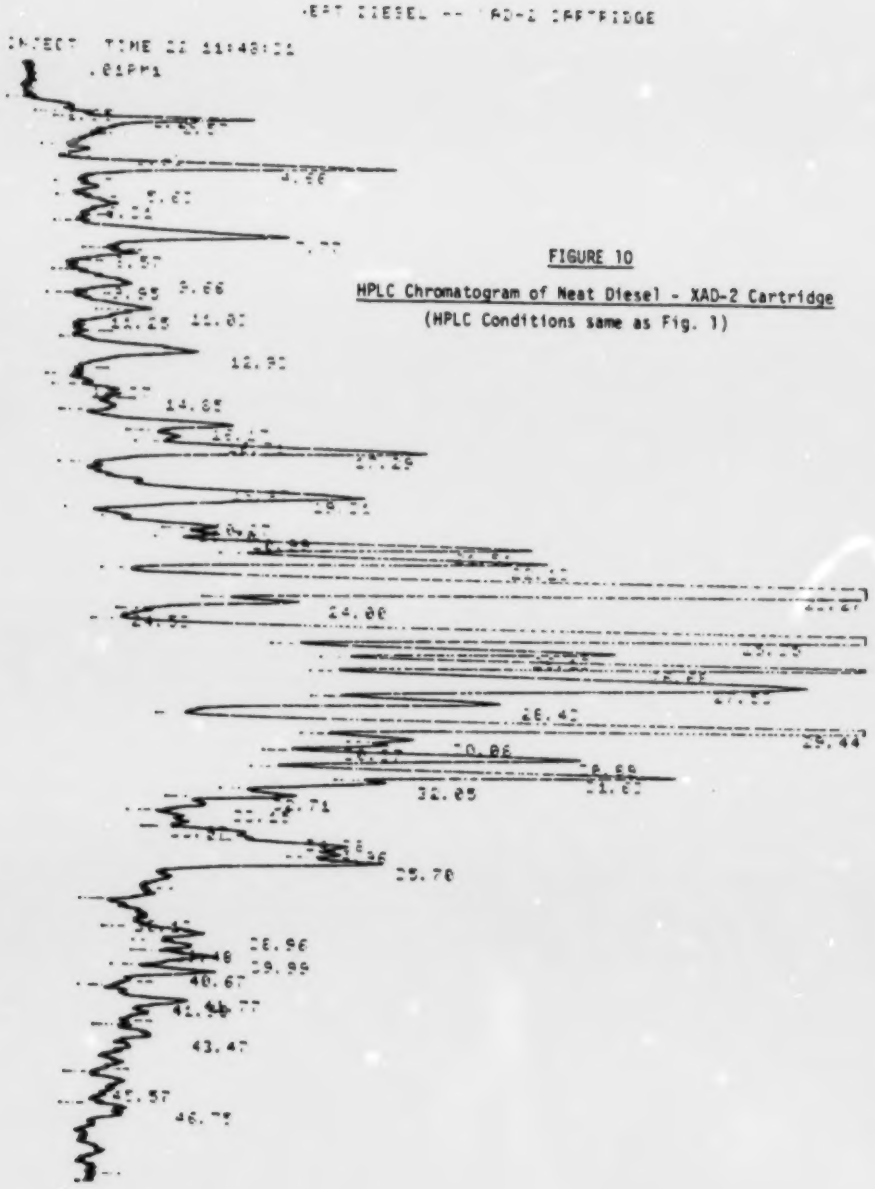


FIGURE 10

HPLC Chromatogram of Neat Diesel - XAD-2 Cartridge

(HPLC Conditions same as Fig. 1)

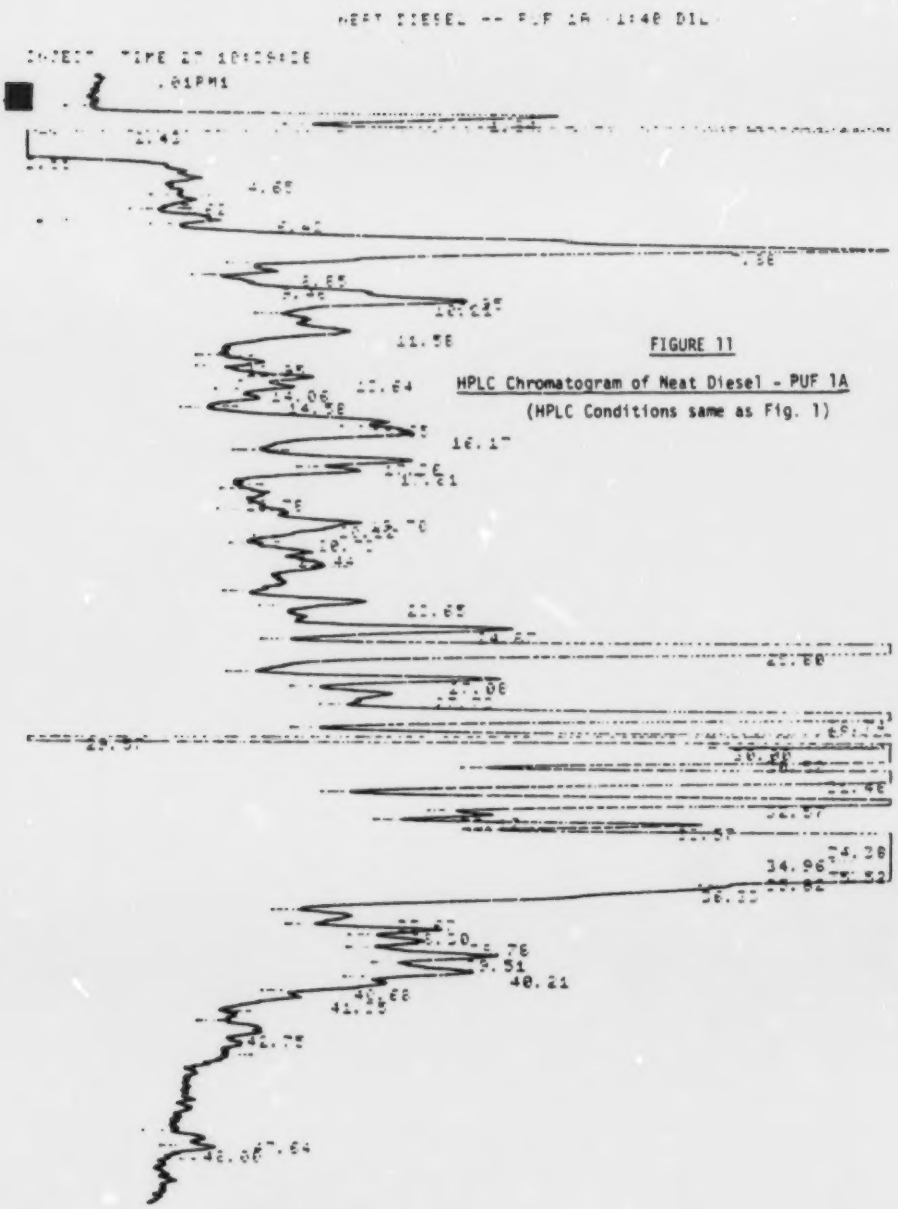


FIGURE 11

HPLC Chromatogram of Neat Diesel - PUF 1A

(HPLC Conditions same as Fig. 1)

FIGURE 12

HPLC Chromatogram of Neat Diesel - PUF 1B

(HPLC Conditions same as Fig. 1)

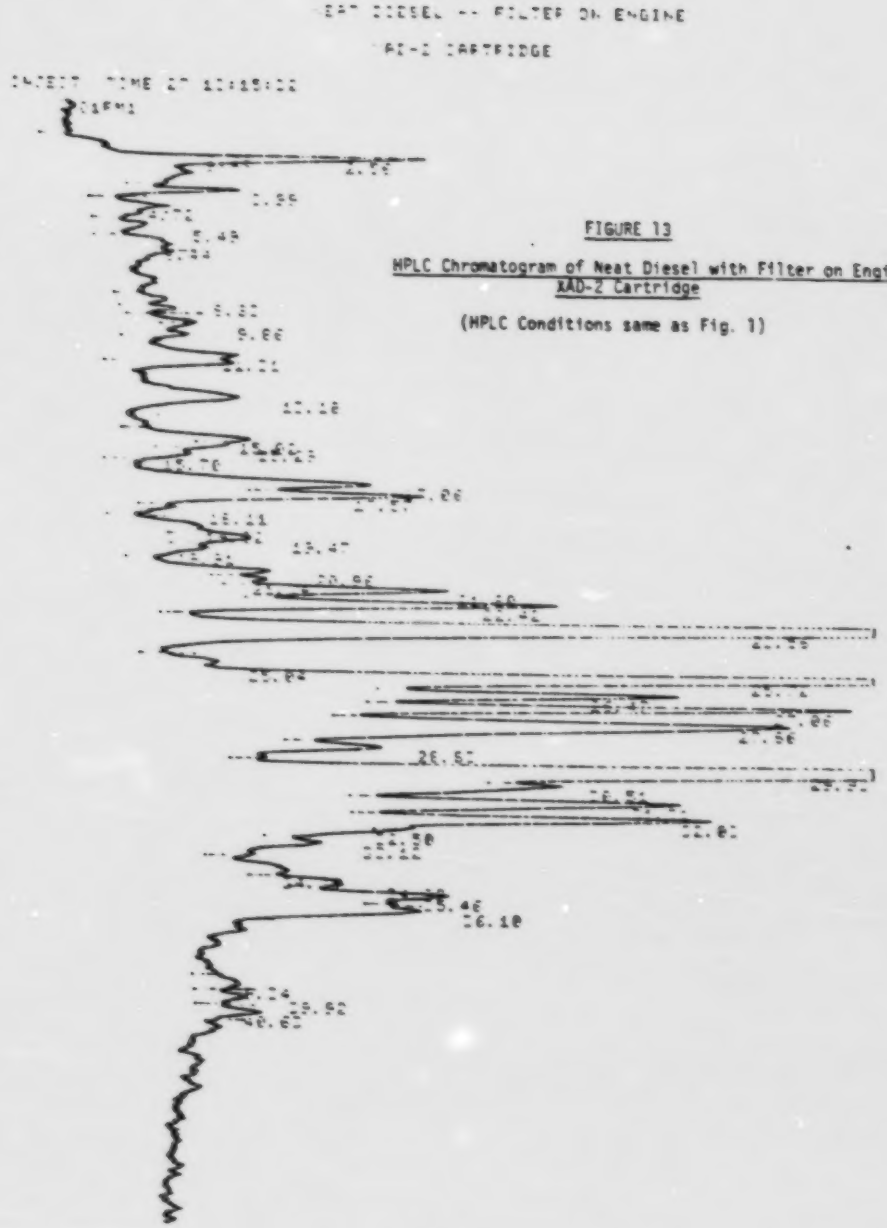


FIGURE 13

HPLC Chromatogram of Neat Diesel with Filter on Engine
XAD-2 Cartridge

(HPLC Conditions same as Fig. 1)

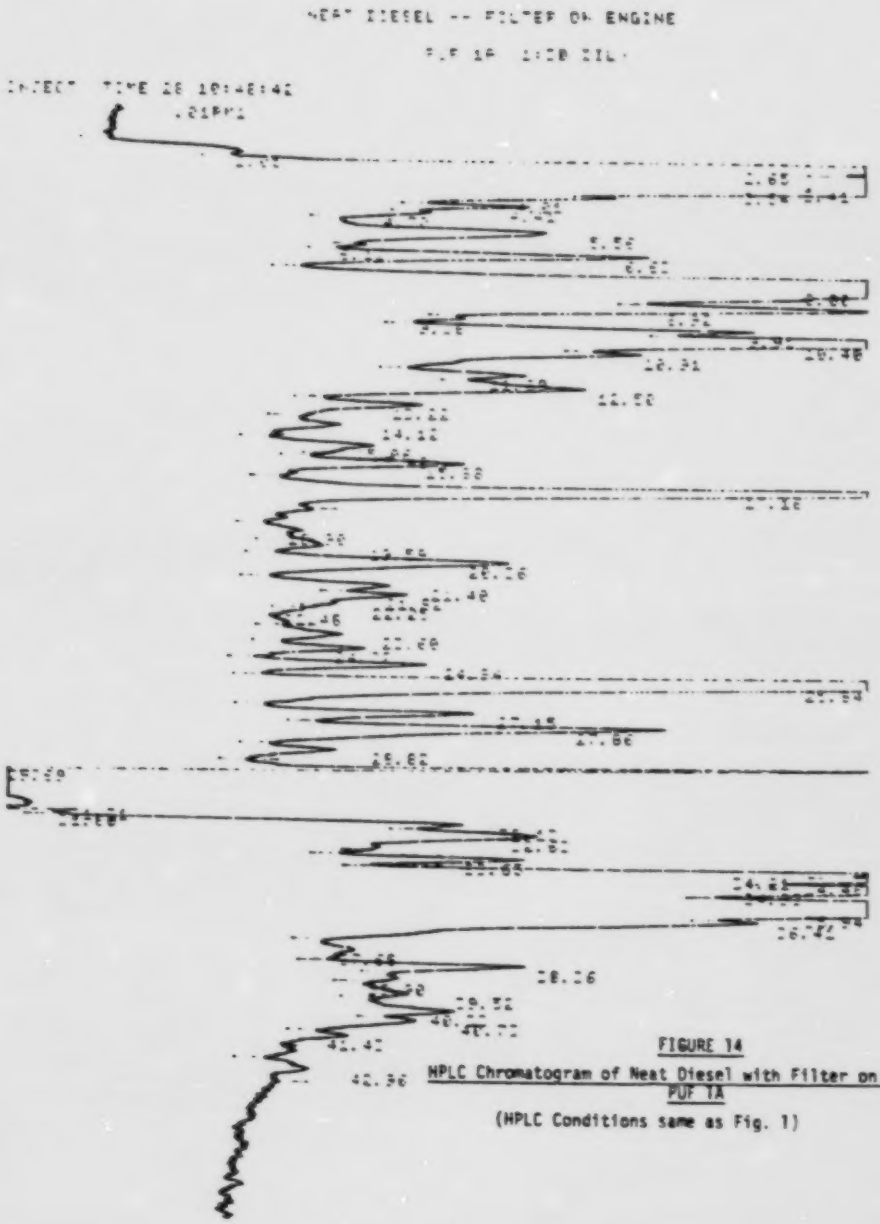


FIGURE 14

HPLC Chromatogram of Neat Diesel with Filter on Engine PUF 1A

(HPLC Conditions same as Fig. 1)

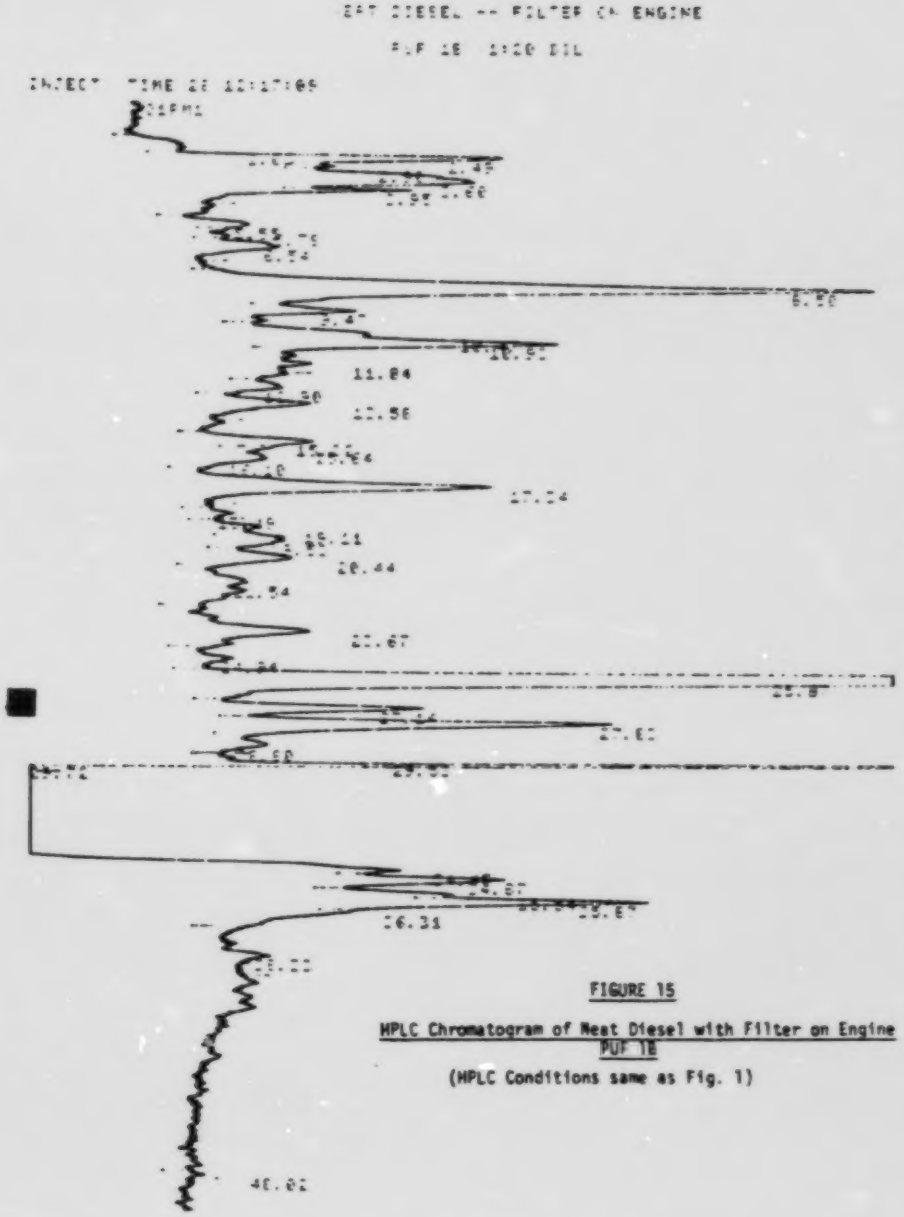


FIGURE 15

HPLC Chromatogram of Neat Diesel with Filter on Engine
PUF 18

(HPLC Conditions same as Fig. 1)

CORRECTED
3-9169

ONTARIO RESEARCH
FOUNDATION

REQ. NO. 3-9169

CONTRACT REPORT NO. OSQ83-00123

ENGINEERING SCIENCES DIVISION

Centre for Alternative Fuel Utilization

COMPLETION OF PRE-TRIAL PHASE OF DIESEL EMISSIONS REDUCTION OPTION DEVELOPMENT

Final Report No. P-4418
ES/CAFU/84-36

Submitted to:

Supply & Services Canada
Science Procurement Branch
Place du Portage, Phase III
11 Laurier Street
Hull, Quebec
K1A 0S5

Attention: Dr. R.G. Agarwal

B. Manicom
W. Robinson
H. Vergeer

September 11, 1984

ONTARIO RESEARCH FOUNDATION

SHERIDAN PARK RESEARCH COMMUNITY

MISSISSAUGA, ONTARIO, CANADA L5K 1B3 • (416) 822-4111 • TELEX 06-982311

76 pp

MICROMEDIA
DEC 19 1994

WE, THE ONTARIO RESEARCH FOUNDATION, STIPULATE THAT THIS DOCUMENT IS SUBJECT TO THE FOLLOWING TERMS AND CONDITIONS

1. ANY PROPOSAL CONTAINED HEREIN WAS PREPARED FOR THE CONSIDERATION OF THE ADDRESSEE ONLY. ITS CONTENTS MAY NOT BE USED BY NOR DISCLOSED TO ANY OTHER PARTY WITHOUT OUR PRIOR WRITTEN CONSENT.
2. ANY TESTING, INSPECTION OR INVESTIGATION PERFORMED BY US WILL BE CONDUCTED IN ACCORDANCE WITH NORMAL PROFESSIONAL STANDARDS. NEITHER WE NOR OUR EMPLOYEES SHALL BE RESPONSIBLE FOR ANY LOSS OR DAMAGE RESULTING DIRECTLY OR INDIRECTLY FROM ANY DEFAULT, ERROR OR OMISSION.
3. ANY REPORT, PROPOSAL OR QUOTATION PREPARED BY US REFERS ONLY TO THE PARTICULAR MATERIAL, INSTRUMENT OR OTHER SUBJECT REFERRED TO IN IT. NO REPRESENTATION IS MADE THAT SIMILAR ARTICLES WILL BE OF LIKE QUALITY.
4. NO REPORT ISSUED BY US SHALL BE PUBLISHED IN WHOLE OR IN PART WITHOUT OUR PRIOR WRITTEN CONSENT.
5. OUR NAME SHALL NOT BE USED IN ANY WAY IN CONNECTION WITH THE SALE, OFFER OR ADVERTISEMENT OF ANY ARTICLE, PROCESS OR SERVICE.
6. WE RESERVE THE RIGHT NOT TO COMMENCE AND/OR CONTINUE ANY WORK UNTIL PAYMENT ARRANGEMENTS SATISFACTORY TO US ARE ESTABLISHED.

TABLE OF CONTENTS

		Page #
1.	SUMMARY	1
2.	INTRODUCTION	2
3.	TEST EQUIPMENT AND PROCEDURES	3
	3.1 Ames Salmonella Mutagenicity Assay	5
	3.2 Gaseous Emissions	5
4.	RESULTS	
	4.1 Test Particulars and Temperature Observations	6
	4.1.1 Burn-off #1	6
	4.1.2 Burn-off #2 and #3	7
	4.1.3 Burn-off #4	
	4.1.4 Particulate Test Results	8
	4.1.5 Gaseous Emissions Summary	9
	4.1.6 Ames Mutagenic Assay Results	9
	4.1.7 Voltage and Amperage Summary	9
5.	CONCLUSIONS	10

TABLE I - Temperature Summary

TABLE II - DPF Build-up Parameters of Engine Assembly at 2200 RPM/55 HP

TABLE III - Particulate Data

TABLE IV - Ames Activity

FIGURES 1 - 50

APPENDIX - (Figures 1-8)

1. SUMMARY

Initial evaluation has been conducted on the use of heating elements in assisting regeneration of ceramic diesel particulate filters (DPF's). Tests were conducted to determine the particulate concentrations and mutagenicity of the material emitted during forced regneration with an electric heating element.

A heating element rated at 24 volts D.C., 4200 watts was used for the regeneration tests. It was found that artificial regeneration was possible using the heating element in conjunction with a low air stream flow passing over the heater surface.

The heating element was found to effectively regenerate the ceramic filter.

Internal filter temperature data will be assessed by Corning Glass Works (CGW) to determine if regeneration using the faceheater leads to excessive thermal stresses.

The long (≃50 min) regeneration time may be unacceptable from a practical mining operation standpoint. Further efforts should be spent to assess the effectiveness of the faceheater in combination with organo-metallic fuel additives.

ORI

2. INTRODUCTION

Ceramic diesel particulate filters (DPF's) are effective in controlling diesel particulate emissions (typically 85% to 95%). This control efficiency is attractive in light of the continuing efforts to minimize diesel engine emissions in underground mining operations. Previous studies have shown that, in some cases, these filters will automatically regenerate depending on the average exhaust gas temperatures. Fuel additives have been shown to reduce the particulate ignition temperature and thus allows for auto-regeneration of DPF's over lower load factor duty cycles. Catalyzed DPF's also exhibit reduced particulate ignition temperatures. Test results show that diesel powered machines with an average exhaust temperature of >390^{o}C can auto regenerate with either of these two assisted regeneration methods.

Many mining machines have a low load factor and thus have average exhaust gas temperatures well below 390^{o}C. It is evident that other means of promoting DPF regeneration are necessary to improve the overall acceptability of these filters in underground mining operations. Factors which impact the overall acceptability of assisted regeneration methods include:

- should be 'on-board';
- regeneration duration should be short;
- system should be simple and fit neatly into the confines of the mining machine;
- energy requirement should be minimal;
- overall cost (capital and operating) should not be excessive.

This report summarizes the activities and test results of a program task to assess the effectiveness of an electric faceheater for assisted regeneration of DPF's.

3. TEST EQUIPMENT AND PROCEDURES

Ontario Research Foundation was supplied, through CANMET, with Corning ceramic diesel particulate filters (DPF's) from Exhaust Controls of Union, New Jersey. One DPF was mounted with a heating element rated at 24 volts D.C., 4200 watts and supplied by Teledyne Stillman. The element was placed ¼" to ½" from the inlet DPF filter face. Eleven type "K" thermocouples were located at strategic locations within the Corning filter to allow for thermal mapping. Figures 1 and 2 show the location of the thermocouples.

Thermocouples were connected to a Data Translation signal conditioning module with analog to digital conversion using Datametrics A/D convertors. All thermocouple signals were processed using an Apple IIe computer with 128K of memory. A special software program was developed in Assembler and Pascal languages to compute and process the signals. This program scans the thermocouples once every 10 seconds and updates readings when a minimum $\pm 4C^{o}$ temperature change occurs. This feature was implemented to reduce the amount of temperature data storage on disk. If a temperature change of less $\pm 4C^{o}$ occurred then only the time is stored on disk. A separate program was developed to display the temperature profile versus time on a hard copy.

The ceramic DPF was installed on a Deutz F8L-413 F/W diesel engine connected to a Clayton dynamomete. The DPF with thermocouples and heating element was installed on the left exhaust bank of the engine (as viewed from the flywheel end of the engine).

The engine was operated at 2200 RPM with an effective load of 55 HP for DPF build-up. Build-up of the DPF was determined by measuring the differential exhaust backpressures across the DPF. A differential pressure of 51 cm of water was required before burn-off tests could be conducted. The maximum allowable exhaust backpressure for this engine is 100 cm of water. Build-up time for the DPF varied from 11.7 hrs to 14.5 hrs.

Engine temperatures were monitored and recorded during the build-up period using "K" type thermocouples in conjunction with a Doric trendicator and a multipoint selector.

Upon completion of the DPF build-up the ceramic filter was removed from the engine to allow for total emissions sampling during regeneration. The DPF was fitted with appropriate flanges to adapt it into the collection system (see Figure 3). Once mounted into the sampling system, two twelve volt batteries and an ammeter were connected in series with the heating element. Burn-off tests were conducted over a period of 43 to 50 minutes. This was determined by the length of time that the battery was able to retain its charge. The voltage across the batteries and heater element current draw were measured and recorded during all tests.

To increase the thermal conductivity and allow for increased regeneration a low flow rate of ambient air was introduced to the closed end of the filter housing the filter element. This was accomplished by directing and measuring 5 cfm air flow through two temperature compensated dry gas meters set in parallel to each other (as shown in Figure 3).

The exhaust of the DPF was directed through a stainless steel dilution tube that utilized a high volume sampling pump. The sampling system was modified with the addition of a second sampling pump when the need for a higher dilution ratio was observed.

Particulates were collected on a 8" x 10" teflon coated glass fibre filter (type T60A20) followed by two polyurethane foam plugs (PUF's). Before testing, the PUF's were extracted for three separate 8 hour periods. The first extraction required dichloromethane to remove the binding material in the foam. Second and third extractions were performed with cyclohexane to remove interfering contaminants.

3.1 Ames Salmonella Mutagencity Assay

The Particulates collected on the Hi-Vol filters and PUF's were tested for mutagenicity using the Ames Salmonella mutagencity assay. Salmonella tester strain TA-98, in the absence of metabolic activation, was used for the assay. Hi-Vol filters and PUF's were extracted for 16 hours with dichloromethane (DCM). The solvent was then taken to dryness and the weight of the soluble organic fraction (SOF) determined. Samples were then dissolved in dimethyl sulfide (DMSO) at a concentration of 200 µg/mL and submitted for testing in the Ames Salmonella mutagenicity assay. Testing was carried out between 0 and 2000 µg per plate to obtain dose-response data. The specific activity as revertants/µg was then calculated for each of the samples from the linear portion of the dose response curve.

3.2 Gaseous Emissions

Concentrations of total hydrocarbons (THC as CH_4), carbon monoxide (CO), carbon dioxide (CO_2) and oxides of nitrogen (NO_x) were measured and recorded during the burn-off. Raw exhaust concentrations were measured during the first tests, with diluted exhaust concentrations measured for the remaining tests. Emissions were sampled via a heated (190°C) stainless steel sampling line with a portion of the gas being drawn through two heated filters (190°C) and delivered to a THC analyzer (Beckman Model 402). The balance of the sample was directed through a second series of heated filters (190°C) and conditioned by passing it through a Hankinson dryer.

The conditioned sample was delivered, under a controlled flow rate, to a CO analyzer (Beckman Model 867), NO_x analyzer (Beckman Model 951A). A separate sample line directed sample gas through a heat exchanger and water trap before being delivered to a CO_2 analyzer (Beckman Model 864).

4. RESULTS

4.1 Test Particulars and Temperature Observations

Table I summarizes the initial test temperature and peak DPF temperatures detected during the tests. The DPF build-up parameters are shown in Table II. All traps were built up at 2200 RPM and 55 HP.

4.1.1 Burn-off #1

After DPF build-up was completed the filter was allowed to cool to room temperature over a 24 hour period. The DPF was then removed from the engine and mounted into the sampling system. Base DPF temperature was approximately 23°C for the start of this burn-off that lasted for 43 minutes.

With the low base DPF temperature, combustion of particulates was complete directly downstream of the heater element coils. Figure 4 illustrates this clearly. The temperature traces for burn-off #1 (Figures 7 to 12) at the heater end of the trap give the same indication. Thermocouples 1,5 and 6, which are at the centre and edge of the trap (hence not in direct line with the heater elements), show a relatively flat temperature profile, peaking at 350°C to 400°C. Thermocouples 3 and 4 however, directly behind the heater element, have peaks of 600°C and 800°C. Figures 13 to 17 (thermocouples located halfway through the traps as shown in (Figure 1) showed the highest temperature at the centre of the trap

(T_1'), and gradually decreasing temperatures toward the centre edge T_2' and T_3'. This illustrates that hot spots were occurring in different areas of the trap when starting regeneration with a cool base temperature.

4.1.2 Burn-off #2 and #3

For these tests the DPF was wrapped with Kao-wool insulation to reduce heat loss and maintain a higher DPF internal temperature prior to initiating the burn-off. The DPF temperature was increased before testing by operating the engine at 2200 RPM at 125 HP. Exhaust temperature was raised to approximately 390°C at this condition. For test #2 the engine was operated for 5 minutes at this setting and 10 minutes for test #3. For both tests the DPF was mounted in the sampling system and burn-off initiated within 30 minutes of shutting the engine down. Burn-off periods lasted 45 minutes for test #2 and 50 minutes for test #3. The initial DPF temperature ranged from 230°C to 400°C for both tests depending on the location of the thermocouples in the filter.

These two tests showed basically the same temperature profiles with a few exceptions. Figure 5 shows that the higher base temperature provided for a more complete regeneration at the front end of the DPF. The temperature profiles are shown in Figures 18 to 29 for burn #2 and Figures 30 to 41 for burn #3.

With the high base DPF temperature condition the temperature was observed to peak for thermocouples 1 to 4 (front of trap) about 10-20 minutes into the test. All four thermocouples had peaks or plateaus >550°C, therefore thermal stresses should not be as great as with test #1. At the middle of the trap the inner 4 temperatures (T_1' to T_4') are relatively similar with the peaks occurring a little later, suggesting the advance of a combustion front. The outer trap temperatures are not

similar however. Figures 22, 23 and 28 (burn #2) show that a combustion front reached T_5 at ≃20 minutes, T_6 at ≃22 minutes and T_5' at ≃25 minutes into the test. In burn #3 there is evidence that the combustion started at T_1' to T_4' a few minutes later than at the front of the trap. However, as can be seen in Figures 34, 35, 40 and 41, there is no evidence of the combustion front reaching the outer part of the trap.

4.1.3 Burn-off #4

With the DPF still insulated, the DPF was removed immediately after build-up at an engine setting of 2200 RPM at 55 HP. At the time of the engine shut down the temperature of the exhaust gases was recorded at 246°C. The initial DPF temperature, measured before regeneration, ranged from 150°C to 220°C.

The dilution tube temperature was reduced for this burn-off by modifying the sampling system to fit in two high-volume sampling pumps. This increased the dilution air flowrate and dilution ratio during the test.

The burn-off pattern on the filter face was similar to burn #1 with the soot burned off only behind the heater element. However, due to an electrical problem in the measurement system, the thermocouple data was not useable.

4.1.4 Particulate Test Results

Analysis of the Hi-Vol filters and PUF's for the soluble and insoluble fractions of the total particulates collected is summarized in Table III. For the first three tests, solubles made up 30.1% to 37.7% of the total particulates. It was found that 78% and 81% of the solubles were collected on the PUF's with 77% and 79% insolubles collected on the 8" x

10" collection filter for tests 2 and 3. The high pass-through of solubles may be attributable to the high dilution tube temperatures.

The solubles collected during the fourth test accounted for 23% of the total particulates. A reduction of pass-through solubles was found with 61% of the solubles collected on the PUF's. Reduction of pass-through solubles is probably due to lower dilution tube temperatures. A general increase in total particulates can be observed, with the lower initial DPF temperature (Tests 1 and 4) being a possible reason.

During burn #1 PUF's were not utilized in the sampling systems, therefore, the total amount of particulates was not determined. Lower particulate collection on the 8" x 10" filter during Test #1 may also be attributed to the higher dilution tube temperature, suspected pass-through of particulates, and incomplete regeneration as shown in Figure 4.

4.1.5 Gaseous Emissions Summary

From raw emissions data summarized in Figures 42 to 49 and Appendix Figures 1 to 8, it can be observed that the emission peaks occur approximately at the same time as the temperature peaks. There were no observable trends between tests. Carbon dioxide peaks ranged from 2 to 7% when corrected back to undiluted concentrations. Carbon monoxide peaks went off scale in the 1 to 2% range except for test #4 which was measured at 1.4%. Total hydrocarbon peaks ranged from 60 to 280 ppm and the nitrogen oxides ranged from 60 to 140 ppm.

4.1.6 Ames Mutagenic Assay Results

Ames Salmonella mutagenicity assay results are summarized in Table IV. A negative mutagenic response was determined for the particulate fraction collected on the PUF's. An average specific mutagenic activity of 1.37

revertants per microgram of soluble particulate was found on the Hi-Vol filter. This revertant concentration resulted in an average concentration 1f 1716 revertants per cubic metre of gas passed through the DPF's. Test #4 had the lowest revertant concentration (0.49) compared to 1.0 and 2.62 for tests 2 and 3 respectively. This may be due to test #4 having a lower percentage of soluble particulates and starting the burn-off at a lower base temperature.

4.1.7 Voltage and Amperage Summary

Two, twelve volt batteries, connected in series, were utilized as the power source for the heating element. The initial voltage was recorded as 22.8 volts for each test with a current reading of approximately 160 amps. The discharge of the battery was reflected by the gradual decrease of both readings over a period of 35 to 40 minutes as shown in Figure 50. After this time period, the voltage and amperage dropped off quickly. The batteries were recharged before each test.

5. CONCLUSIONS

The heating element proved to be effective in regenerating loaded ceramic traps when the traps were purged flushed with 5 cfm of air.

Starting regeneration with a cool trap (23^{o}C) resulted inhot spots in the traps. When a presoaked hot trap (230^{o}C to 400^{o}C) was regenerated the regeneration was more complete and resulted in reduced high temperature zones.

Regeneration time was approximately 50 minutes based, on a 22.8 VDC at 170 amperes starting condition.

Carbon dioxide, total hydrocarbons and total oxides of nitrogen levels were at lower concentrations, or as low as, normally found in the raw exhaust of the Deutz F8L-413 F/W. Carbon monoxide concentration was found to be 1.4% or higher and is approximately 100 times higher than normal diesel exhaust. Total particulate ranged from 9 to 57 mg/m^3 and is less than the raw engine exhaust concentration. However, the 5 cfm flow rate here is considerably less than the maximum 400 cfm exhaust flow rate of the test engine.

Ames mutagenic activity in raw engine exhaust is approximately ≃14,000 revertants per cubic metre while during the regeneration they were only ≃1,700 revertants per cubic metre; again with a much lower flow rate.

The electric face heater is effective for regeneration of DPF's without causing any significant emissions provided it is performed in a well ventilated area.

H.C. Vergeer
Senior Technologist
Centre for Alternative Fuel Utilization
Engineering Sciences Division

:sd

TABLE I

TEMPERATURE SUMMARY

Burn-off Test No.	Exhaust Gas Temp. Before DPF Removed from Engine (^{o}C)	Initial DPF Internal Temp. for Burn-off (^{o}C)	Max. Temp. of DPF During Burn-off (^{o}C)
1	23	23	>1000
2	390	230-400	~ 900
3	390	230-400	~ 930
4	246	150-250	~ 950

ERRATUM

Contents (Table II: DPF Build-up Parameters of Engine Assembly at 2200 RPM/55 HP)

The following is an explanation of thermocouple locations for temperatures found in Table II. (Locations referenced from flywheel end.)

T1 - intake combustion air

T2 - right exhaust - no DPF

T3 - left wheel - before DPF

T4 - fuel temperature

T5 - cylinder head temperature

T6 - oil temperature

T7 - cooling air intake

T8 - left exhaust - after DPF

TABLE II

DPF BUILD-UP PARAMETERS OF ENGINE ASSEMBLY AT 2200 RPM/55 HP

Test #	Time hrs.	Backpressure (CmH_2O) Before DPF	After DPF	DP	Temperatures ^oC (Locations Reference to Figure) T1	T2	T3	T4	T5	T6	T7	T8
1	0	25	9.5		20	350	375	19	109	101	19	359
	12.75	59	8	51	21	243	247	20	89	92	20	231
2	0	27	8.5		21	240	237	20	88	92	19	230
	12.75	58.5	7.5	51.5	24	254	249	24	91	93	23	247
3	0	11	7.5		22	232	226	18	80	75	20	198
	**14.75	59.5	8.0	51.5	20	236	238	18	83	91	18	235
4	0	17	8.5		24	250	246	19	88	79	18	234
	11.75	59.5	9.0	50.5	27	251	251	27	97	94	29	246

* High temperatures due to engine induced burn-off previous to beginning of build-up.

** Longer build-up time attributed to a leak that was corrected during the early build-up hours.

TABLE III

PARTICULATE DATA

Burn-off Test No.	Insoluble Loading (mg)	(mg/m^3)	% of Total	Soluble Loading (mg)	(mg/m^3)	% of Total	Total (mg)	Concentration (mg/m^3)
1 Hi-Vol Filter	124.3	18.19	67.7	59.3	8.68	32.3	183.6	26.86
Pufs	-	-	-	none used -		-	-	-
2 Hi-Vol Filter	51.1		85.4	8.7		14.6	59.8	
Pufs	15.4		32.8	31.6		67.2	47.0	
Combined	66.5	10.05	62.3	40.3	6.09	37.7	106.8	16.14
3 Hi-Vol Filter	38.2		90.7	3.9		9.3	42.1	
Pufs	10.1		37.4	16.9		62.6	27.0	
Combined	48.3	6.23	69.9	20.8	2.681	30.1	69.1	8.916
4 Hi-Vol Filter	273.3		87.3	39.7		12.7	313.0	
Pufs	70.0		53.0	62.0		47.0	132.0	
Combined	343.3	44.42	77.1	101.7	13.16	22.9	445.0	57.58

ERRATUM

Contents (Table III: Particulate Data)

The loading values (mg/m^3) of the insoluble and soluble particulates were calculated using the corrected volume of air passed through the DPF's via dry gas meters.

TABLE IV

AMES ACTIVITY*

Burn-off Test No.		Specific Activity (revertants/μg)	Mutagen Concentration (revertants/m^3)	Soluble Organic Fraction (mg/m^3)
2	Hi-Vol Filter	1.00	1314	1.3
	Pufs	Negative (non-mutagenic)	Neg.	4.77
3	Hi-Vol Filter	2.62	1318	0.5
	Pufs	Neg.	Neg.	2.17
4	Hi-Vol Filter	0.49	2517	5.1
	Pufs	Neg.	Neg.	8.02

* Note mutagen concentration (rev/m^3) based on air volume supplied to the DPFs (0.14 m^3/min). Ames test not conducted on sample from Test 1.

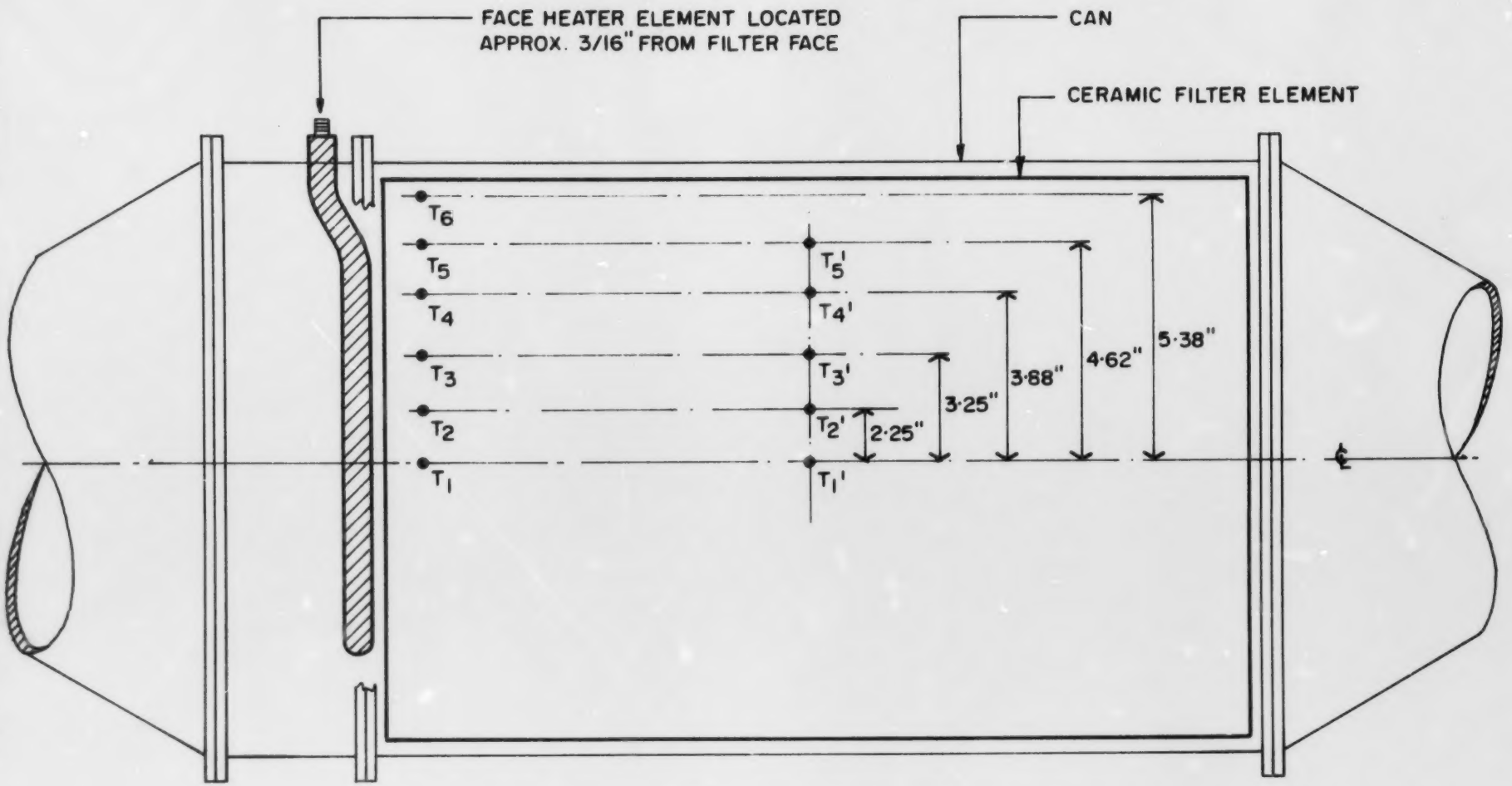


FIGURE 1 SCHEMATIC OF CERAMIC FILTER SHOWING THERMOCOUPLE LOCATIONS

FIGURE 2

View of DPF Outlet Face

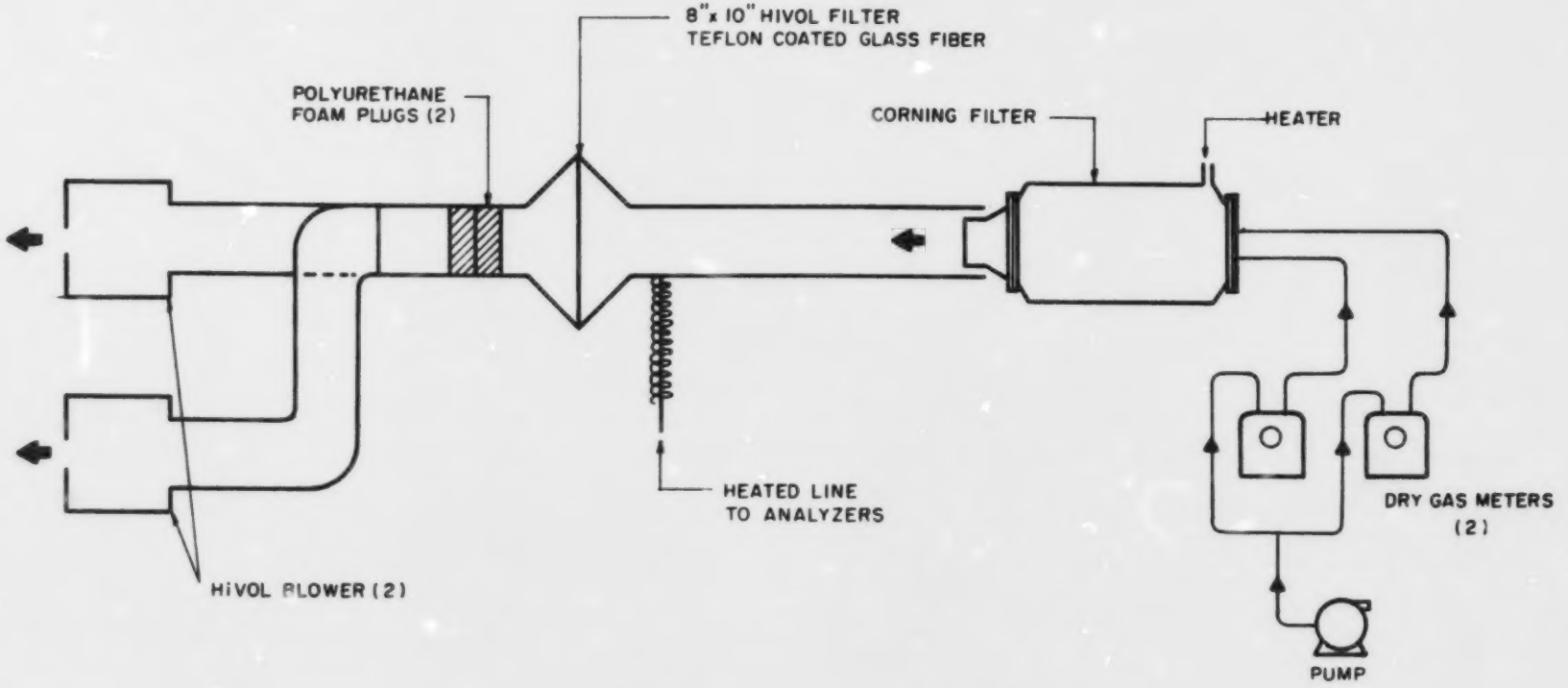


FIGURE 3 MODIFIED DILUTION TUBE SAMPLING SYSTEM SCHEMATIC.

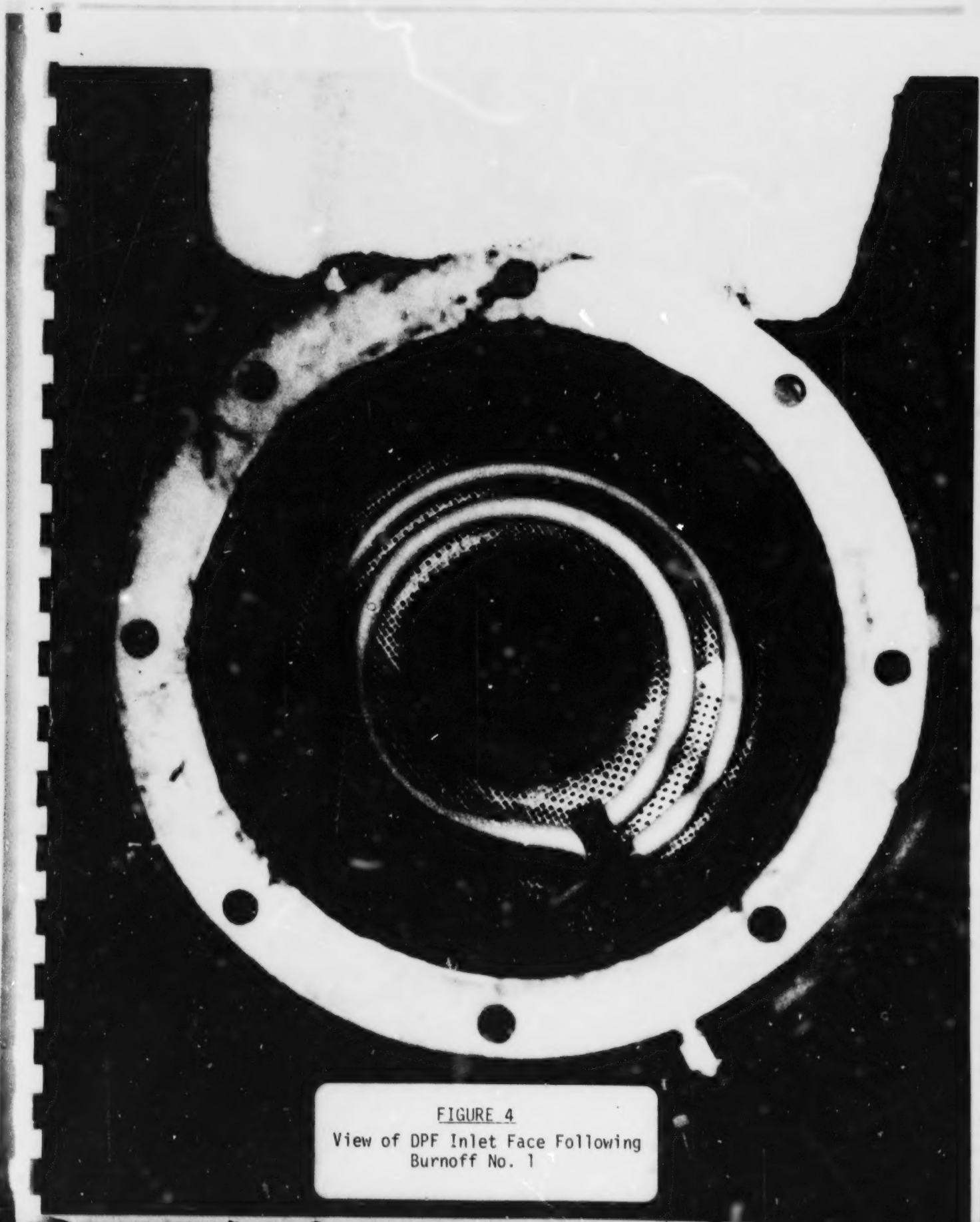


FIGURE 4
View of DPF Inlet Face Following
Burnoff No. 1

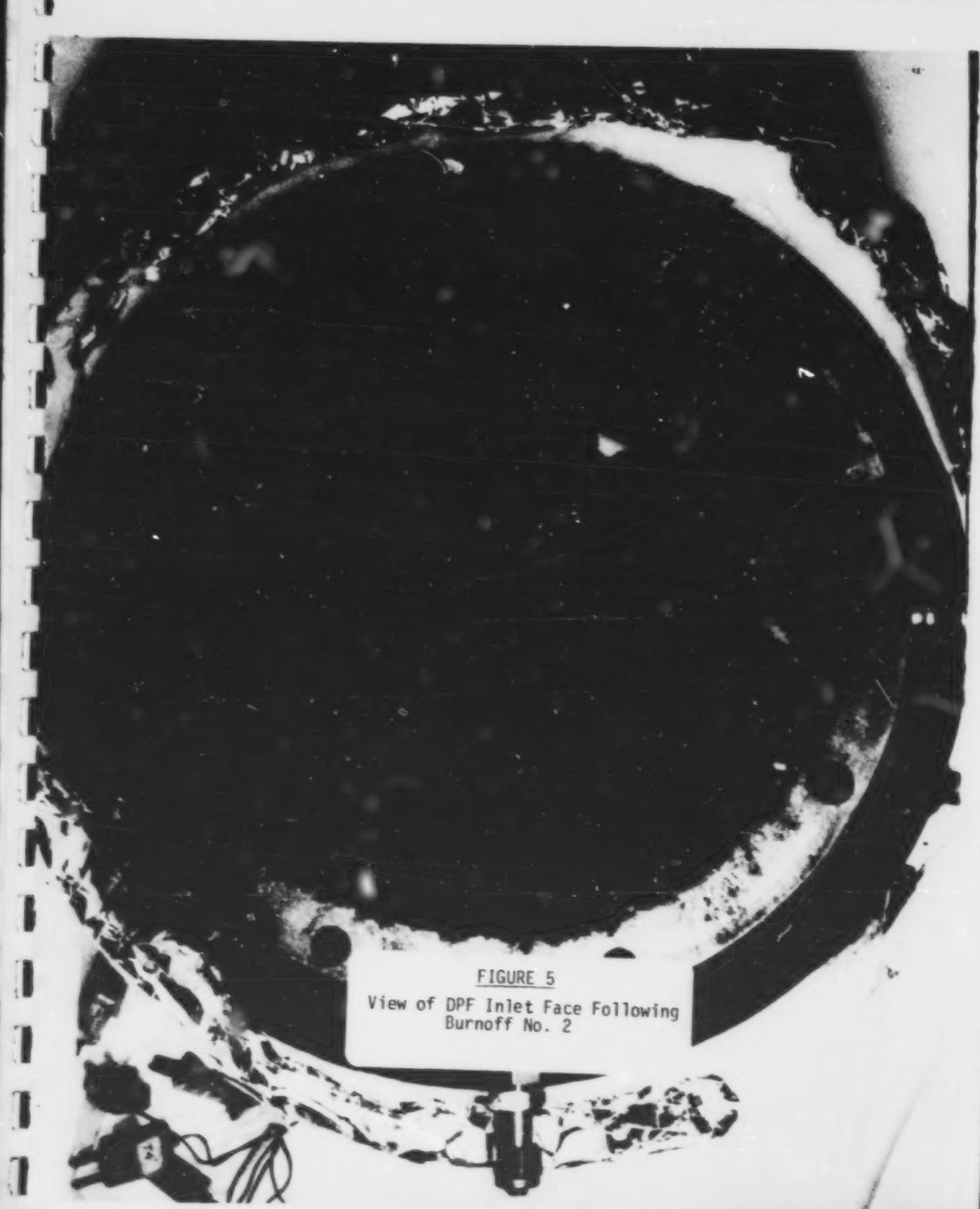


FIGURE 5

View of DPF Inlet Face Following Burnoff No. 2

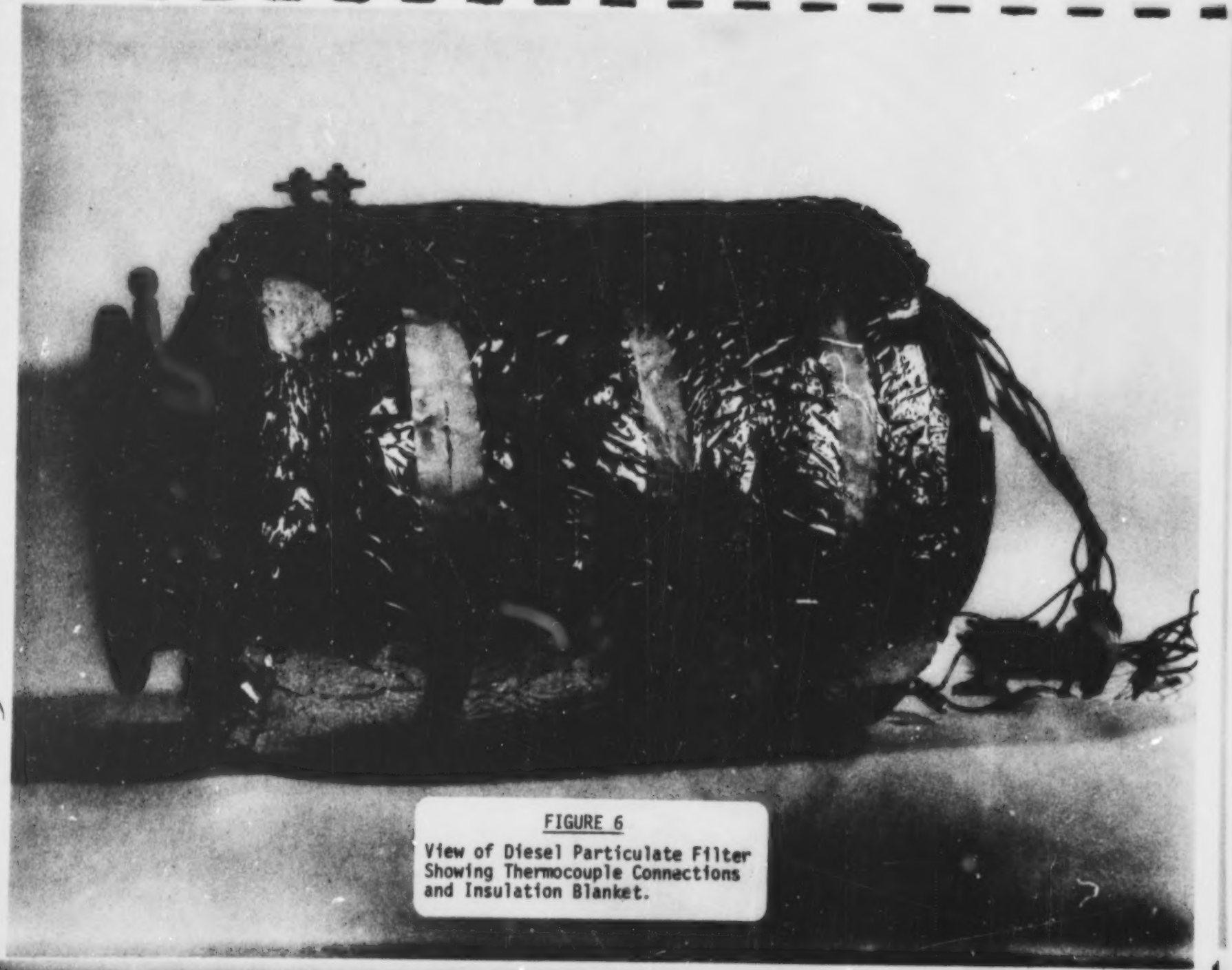


FIGURE 6

View of Diesel Particulate Filter Showing Thermocouple Connections and Insulation Blanket.

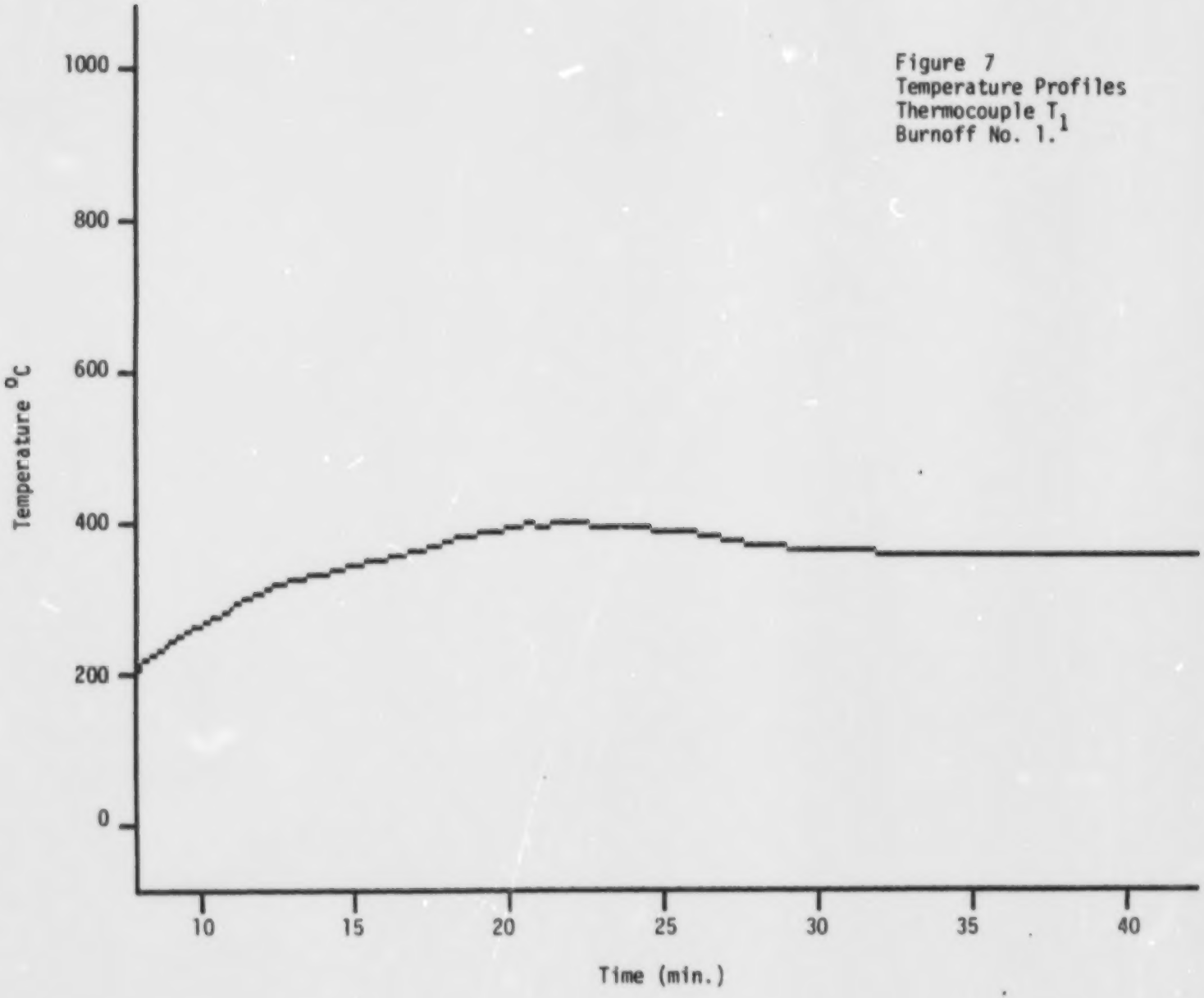


Figure 7
Temperature Profiles
Thermocouple T_1
Burnoff No. 1.

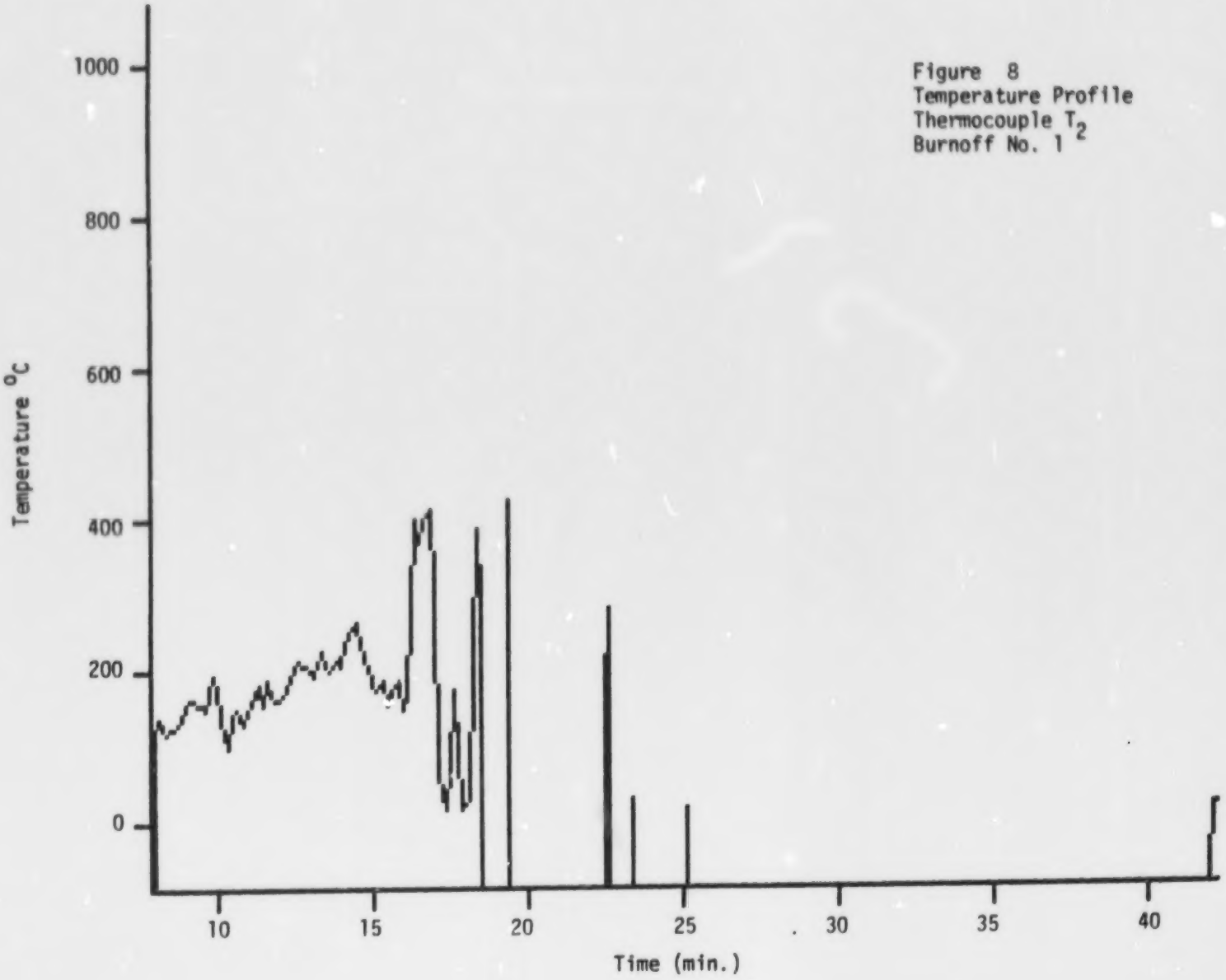


Figure 8
Temperature Profile
Thermocouple T_2
Burnoff No. 1

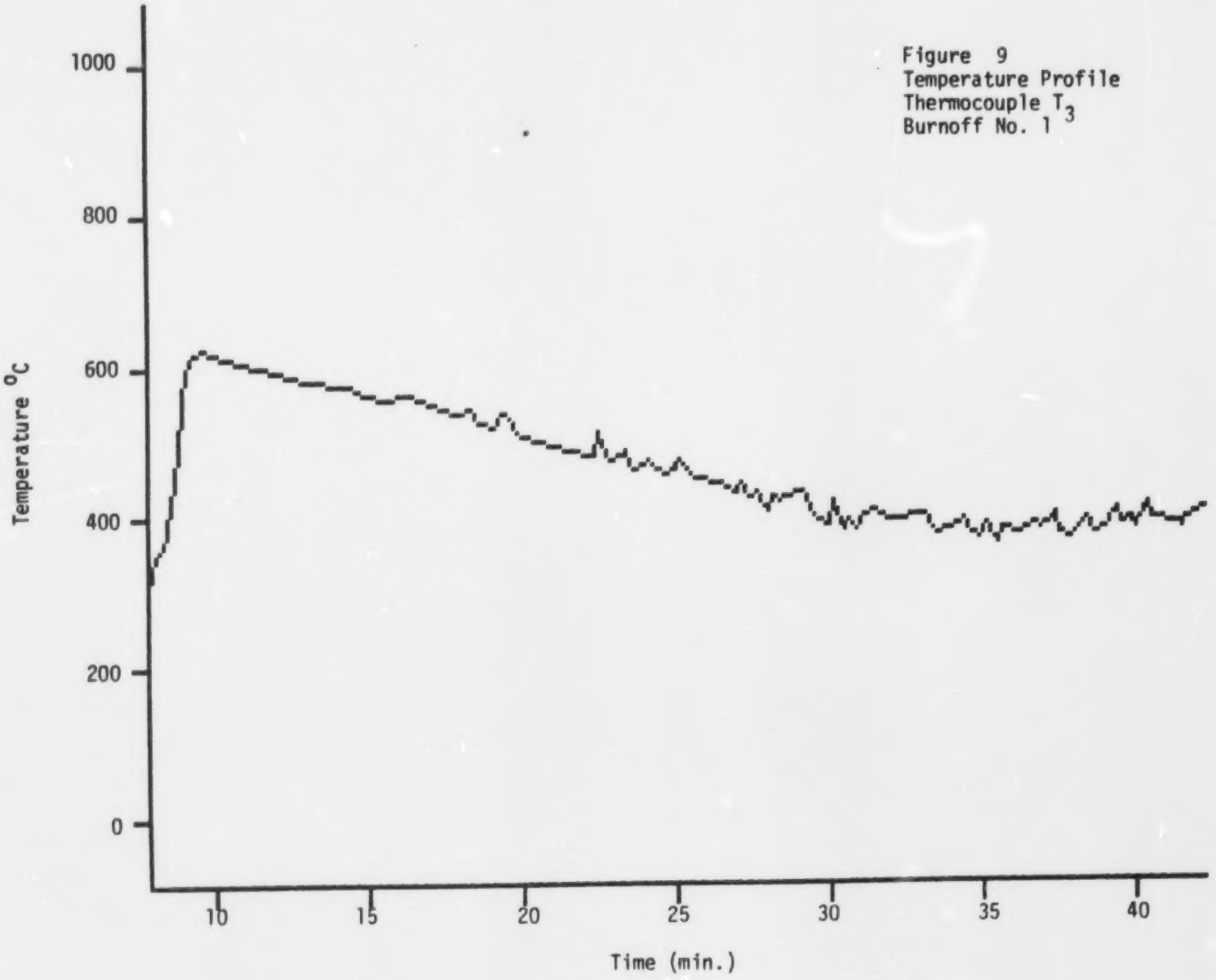


Figure 9
Temperature Profile
Thermocouple T_3
Burnoff No. 1

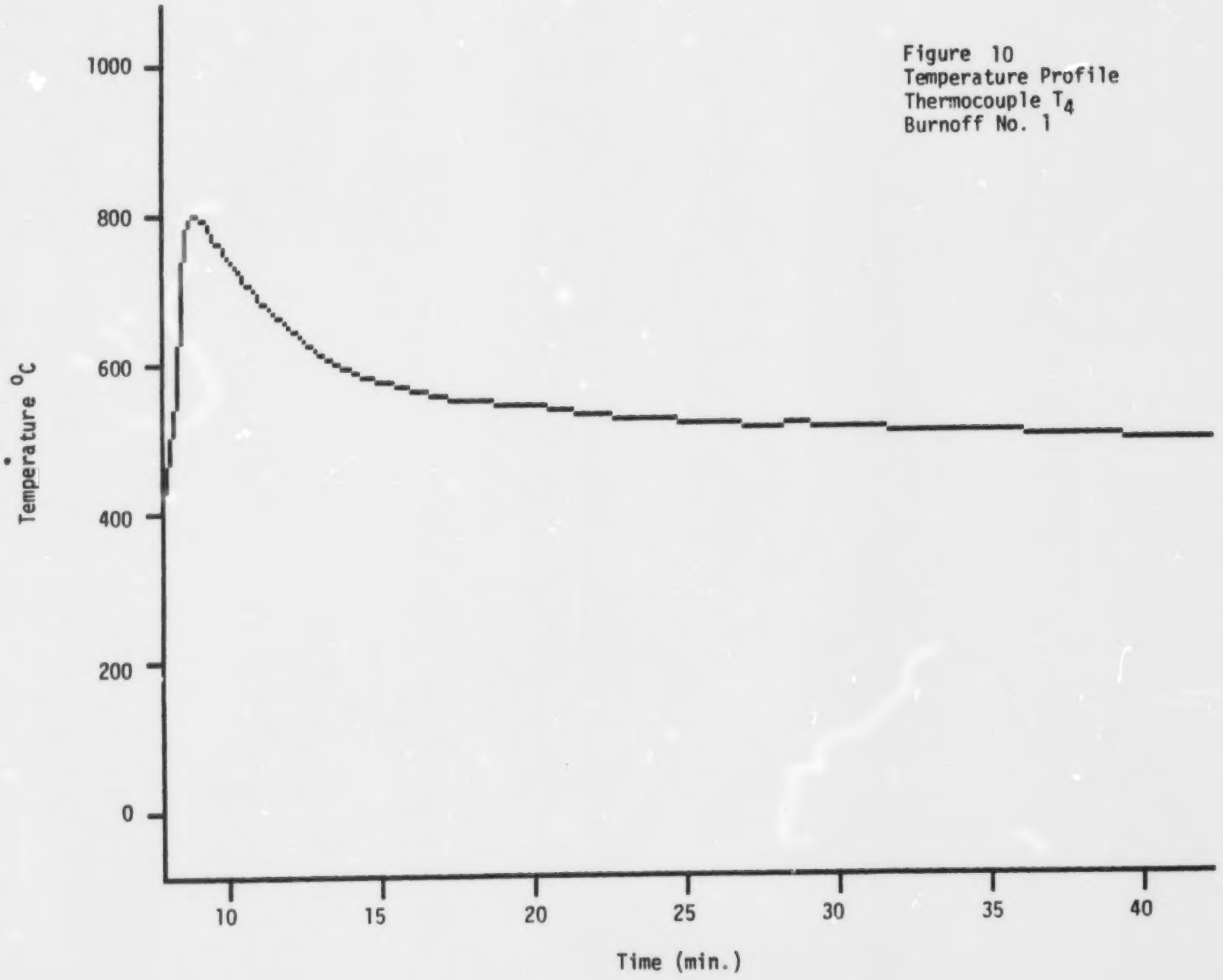


Figure 10
Temperature Profile
Thermocouple T_4
Burnoff No. 1

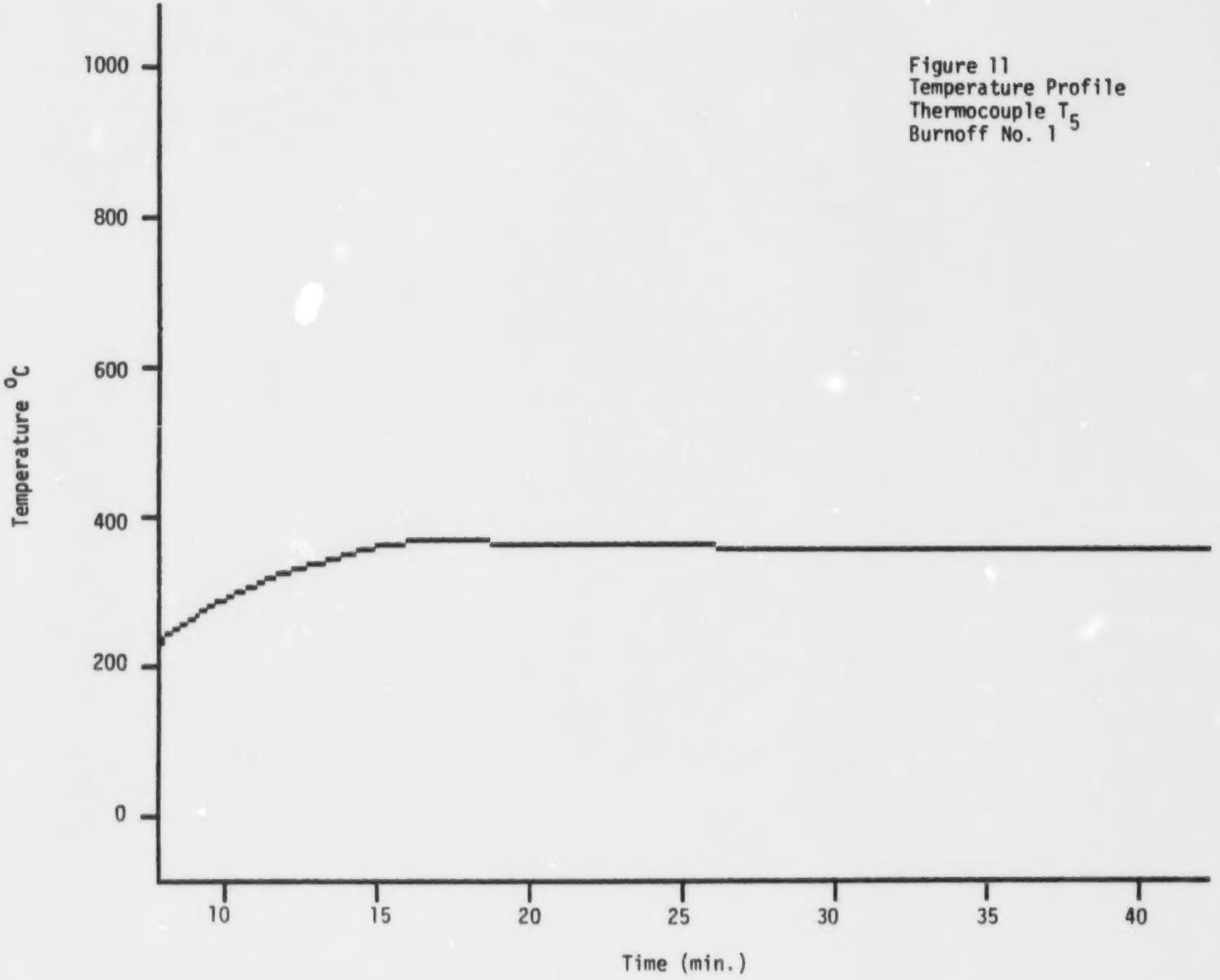


Figure 11
Temperature Profile
Thermocouple T_5
Burnoff No. 1

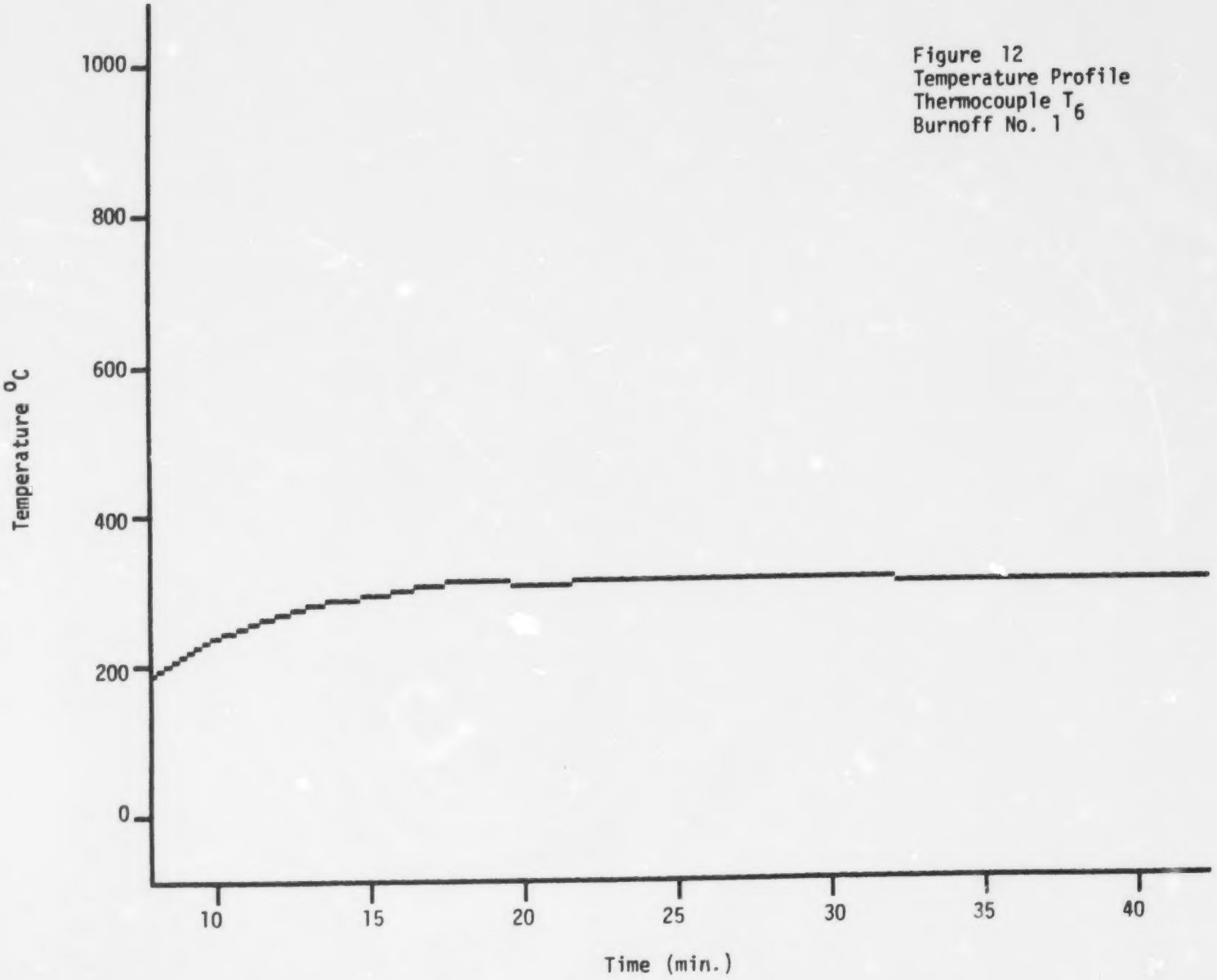


Figure 12
Temperature Profile
Thermocouple T_6
Burnoff No. 1

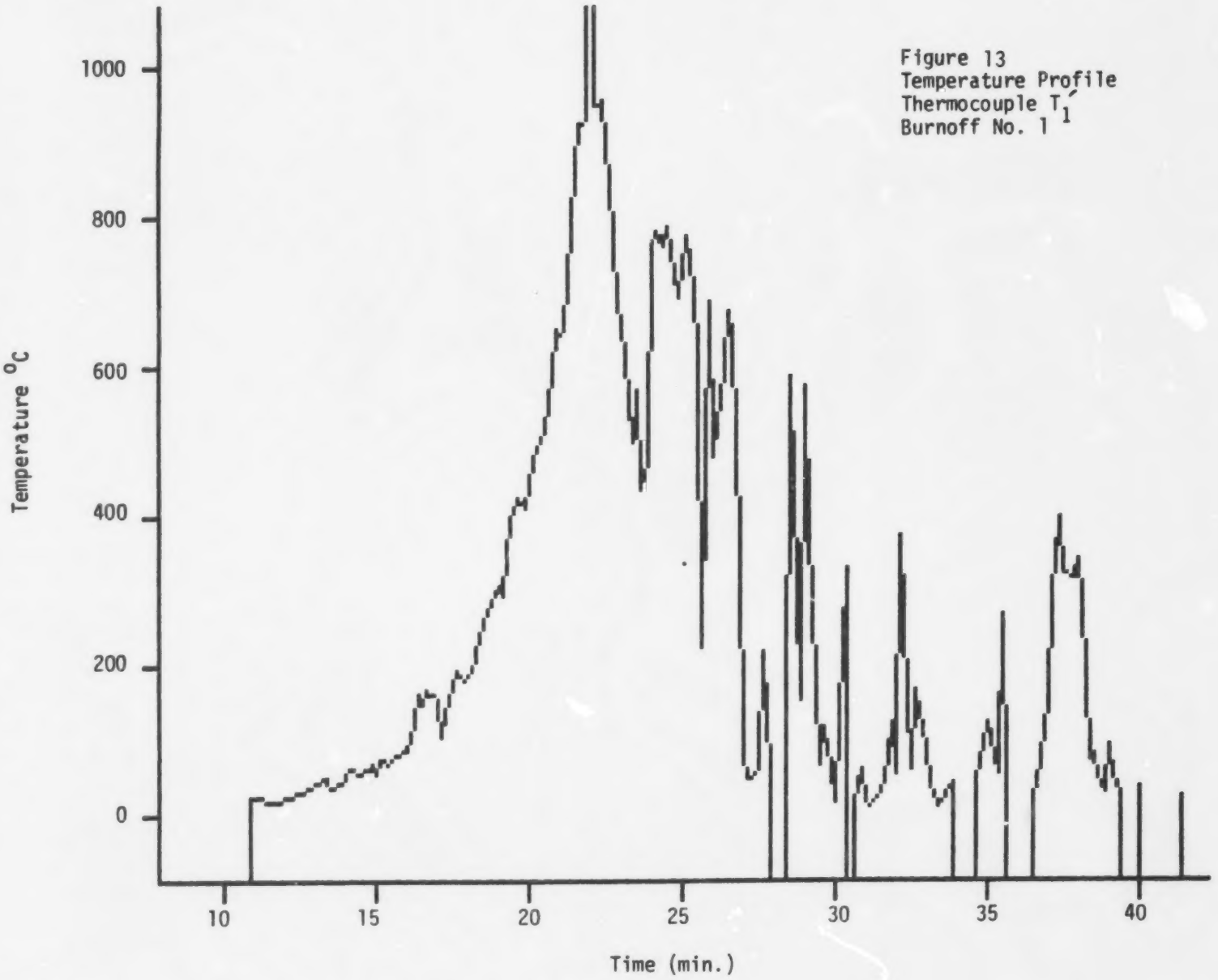


Figure 13
Temperature Profile
Thermocouple T_1'
Burnoff No. 1

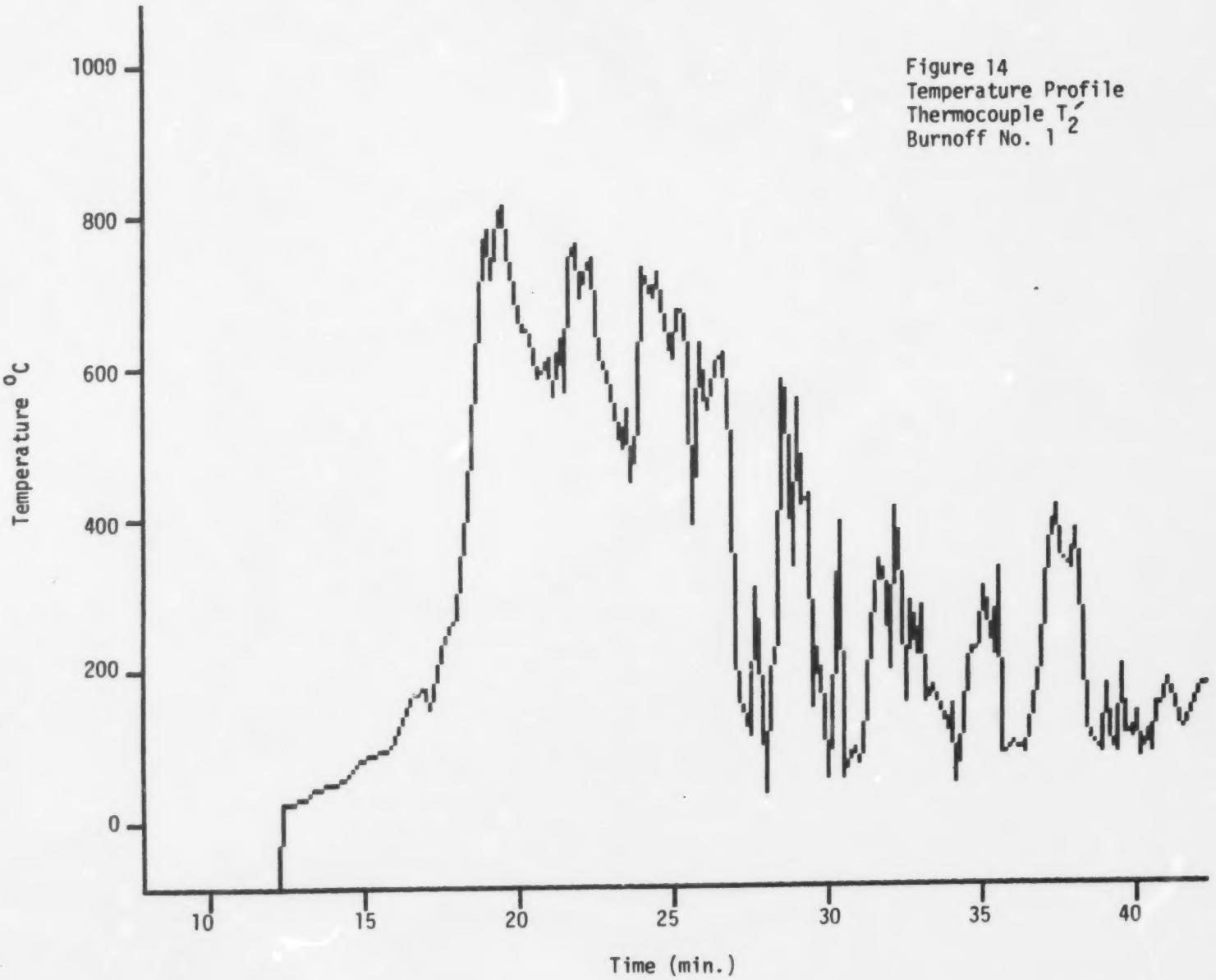


Figure 14
Temperature Profile
Thermocouple T_2'
Burnoff No. 1

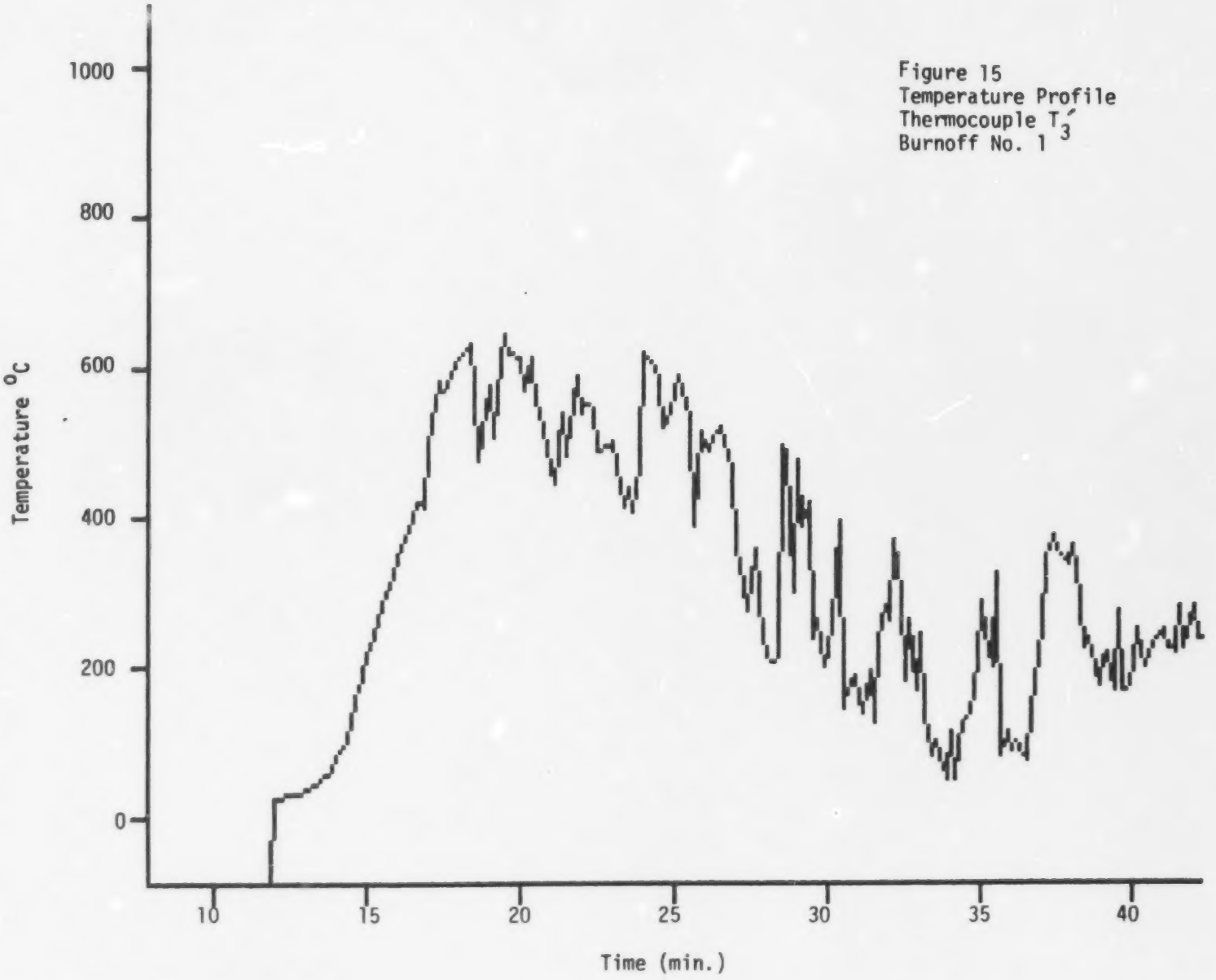


Figure 15
Temperature Profile
Thermocouple T_3'
Burnoff No. 1

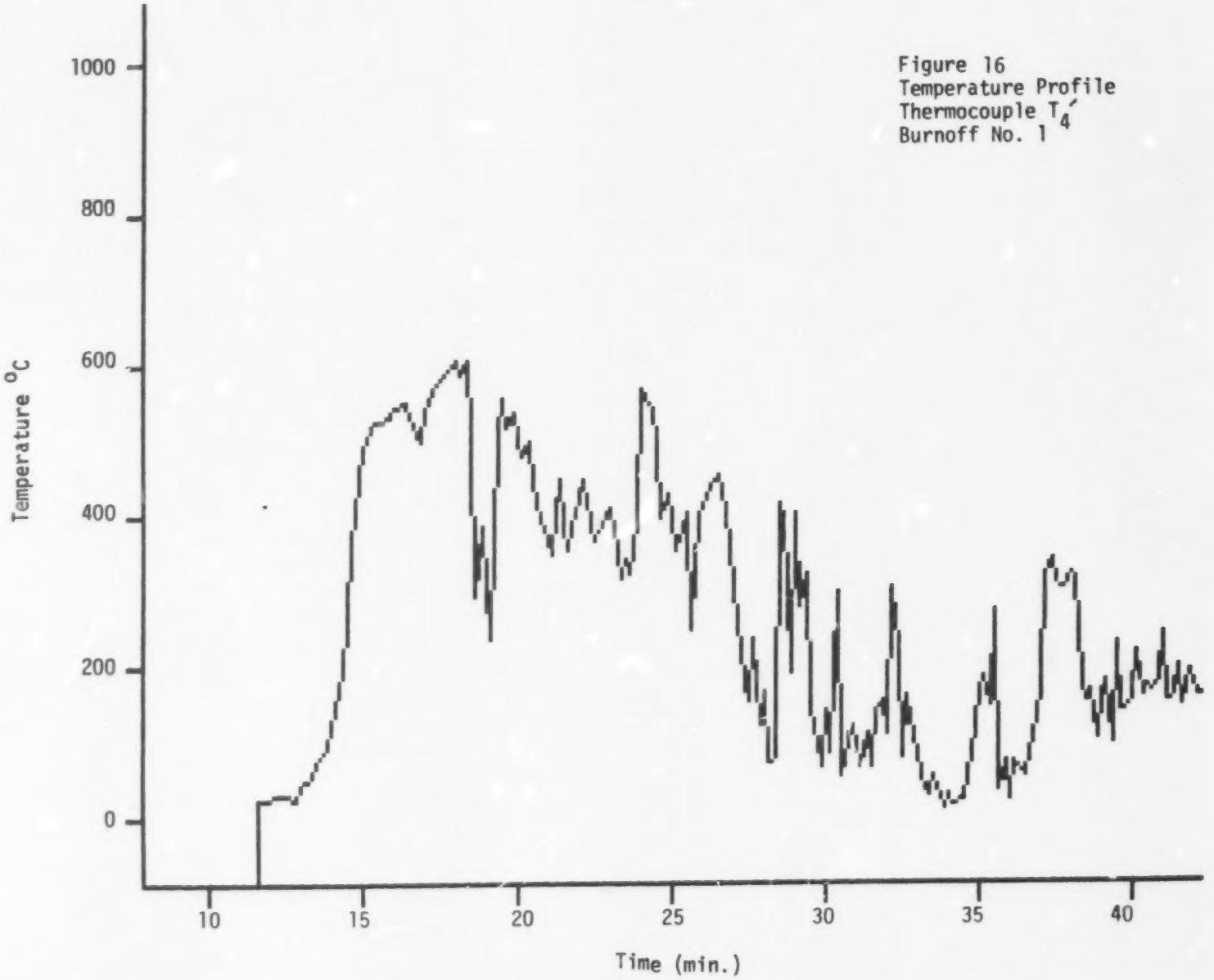


Figure 16
Temperature Profile
Thermocouple T_4'
Burnoff No. 1

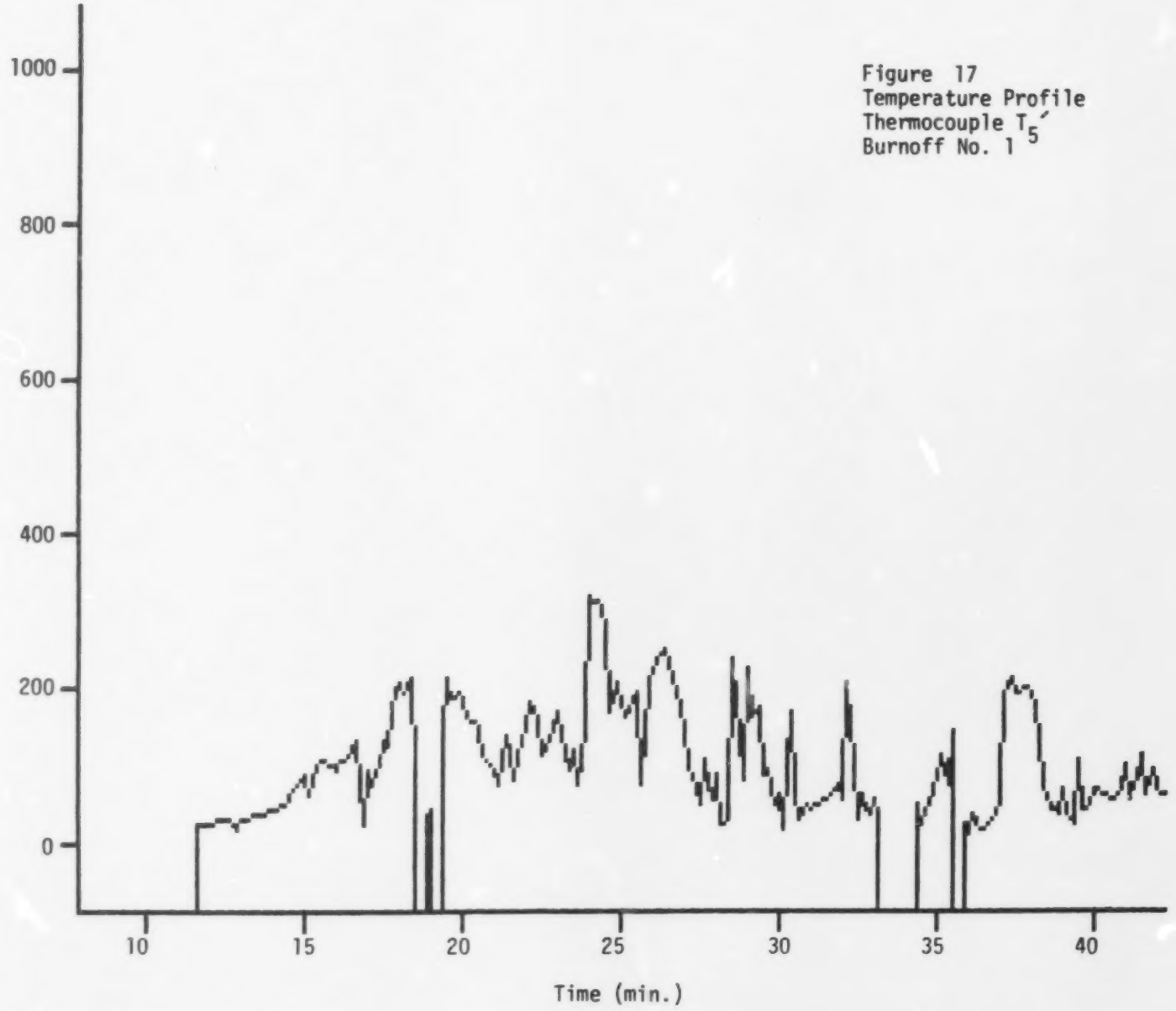


Figure 17
Temperature Profile
Thermocouple T_5'
Burnoff No. 1

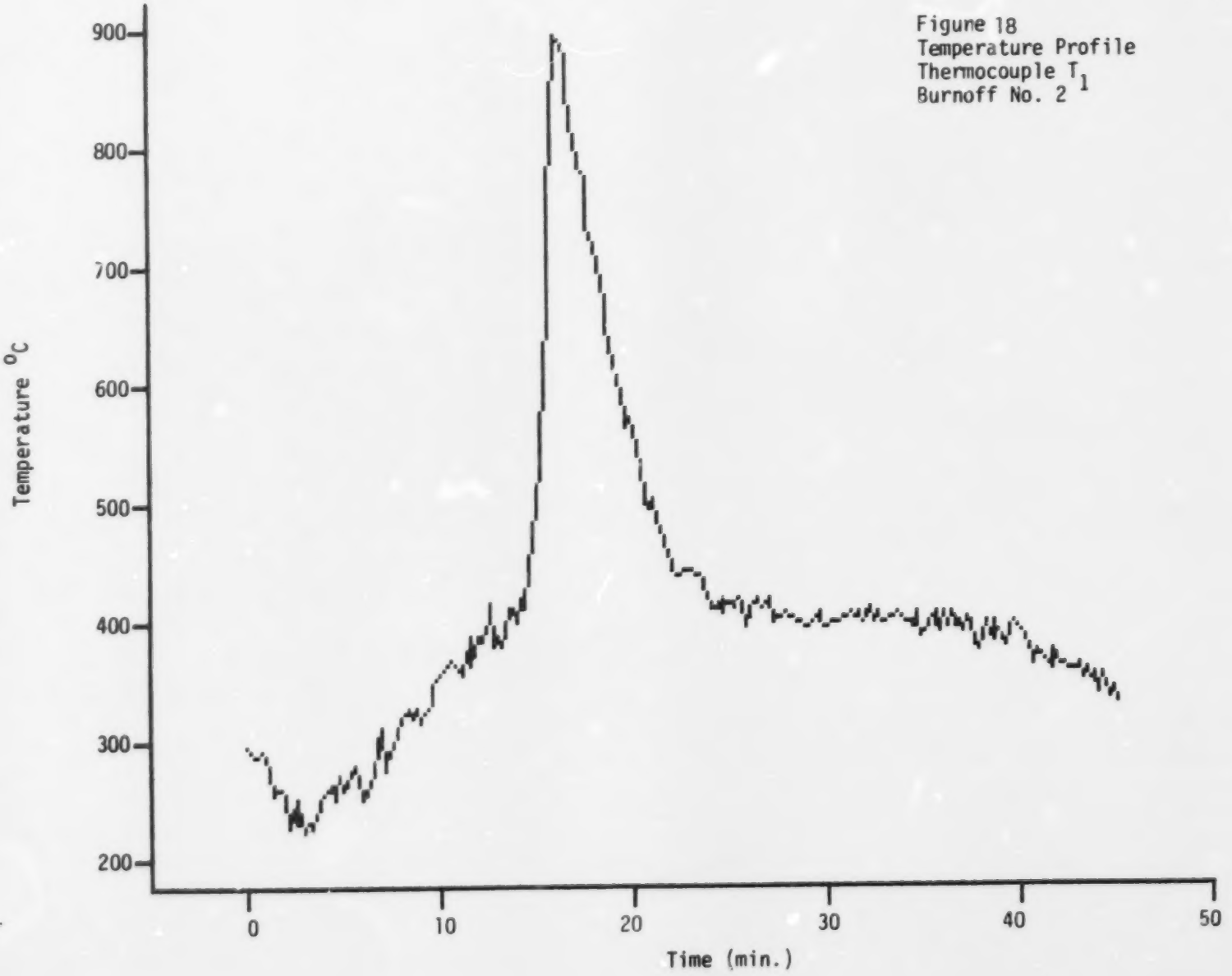


Figure 18
Temperature Profile
Thermocouple T_1
Burnoff No. 2

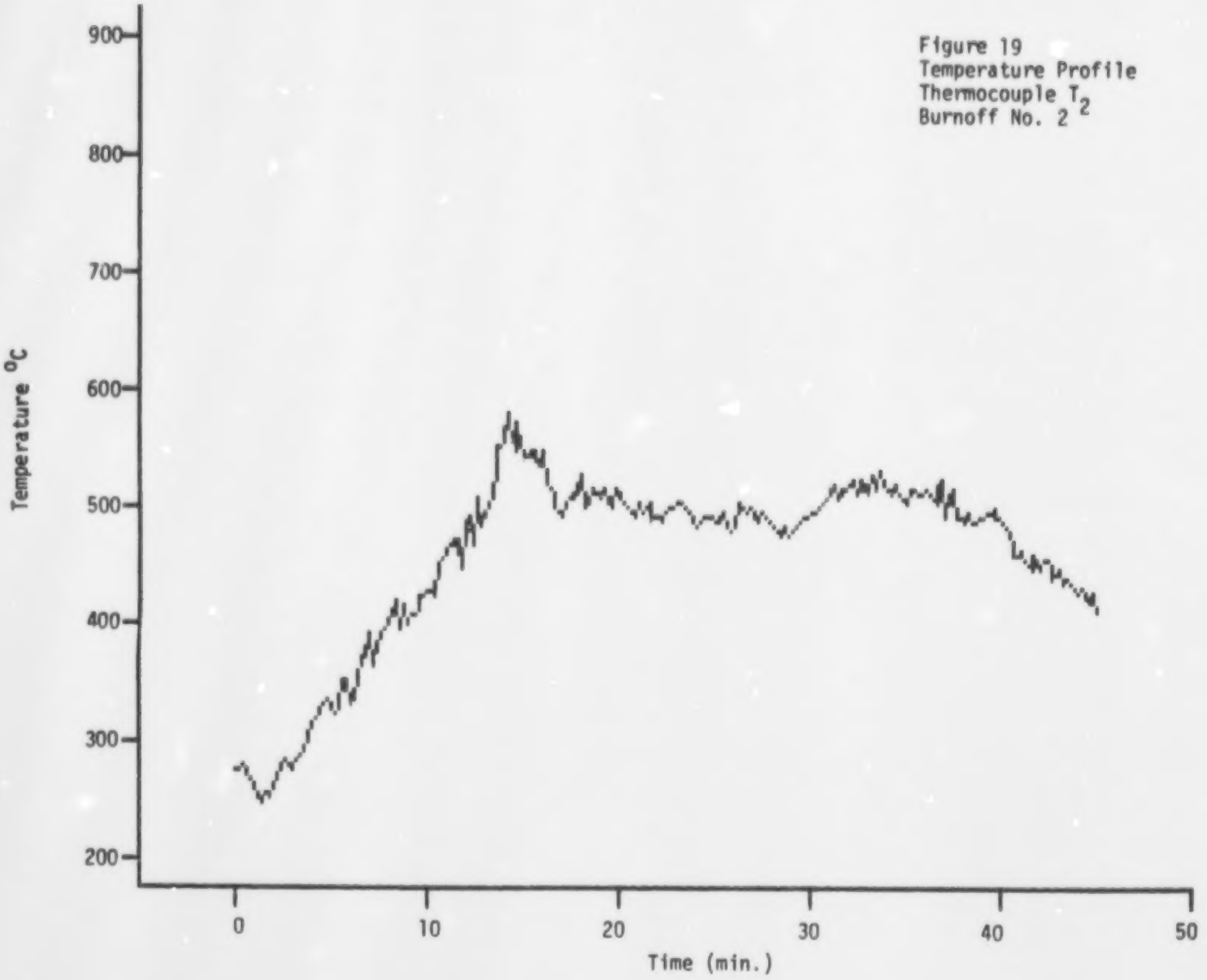


Figure 19
Temperature Profile
Thermocouple T_2
Burnoff No. 2

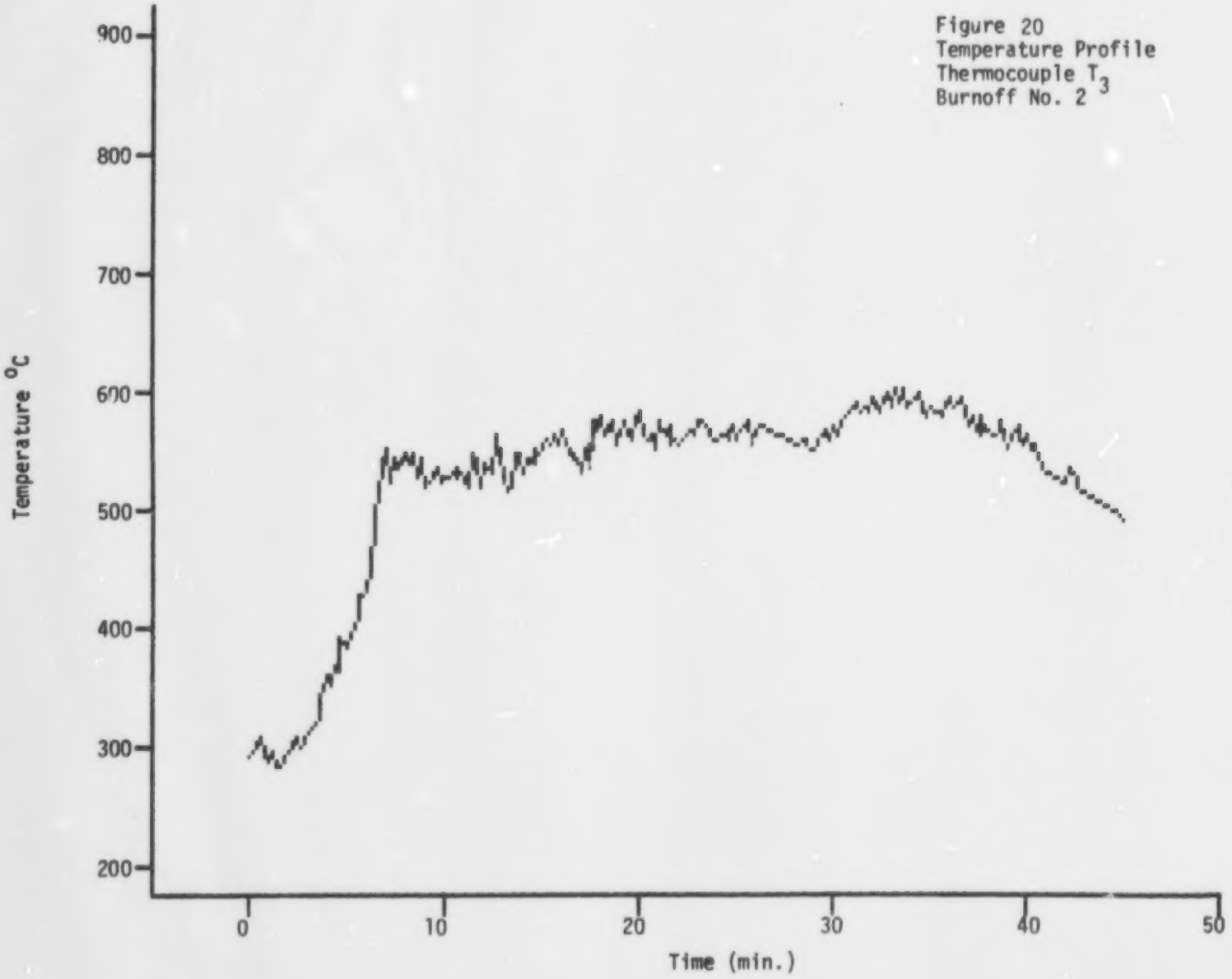


Figure 20
Temperature Profile
Thermocouple T_3
Burnoff No. 2

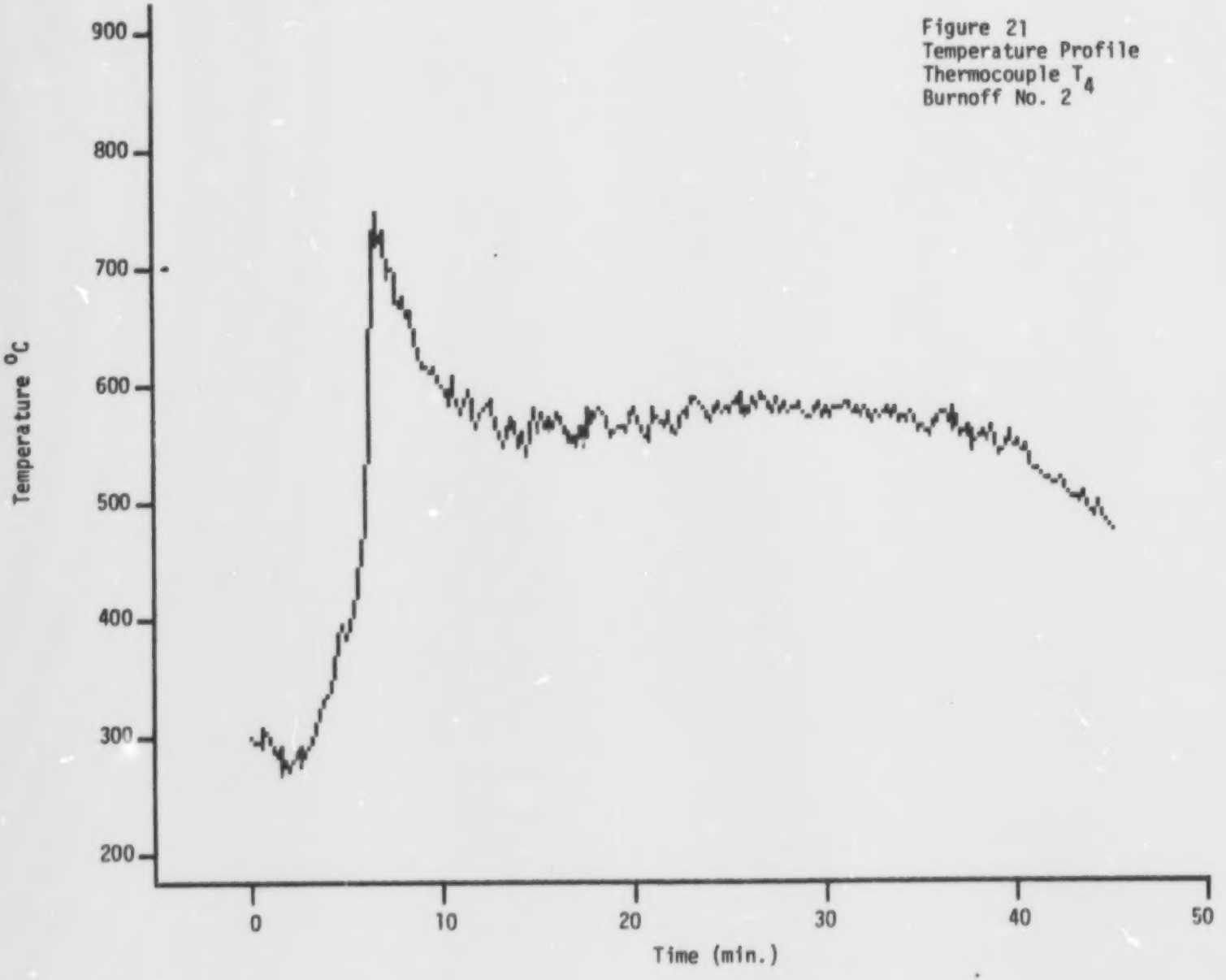


Figure 21
Temperature Profile
Thermocouple T_4
Burnoff No. 2

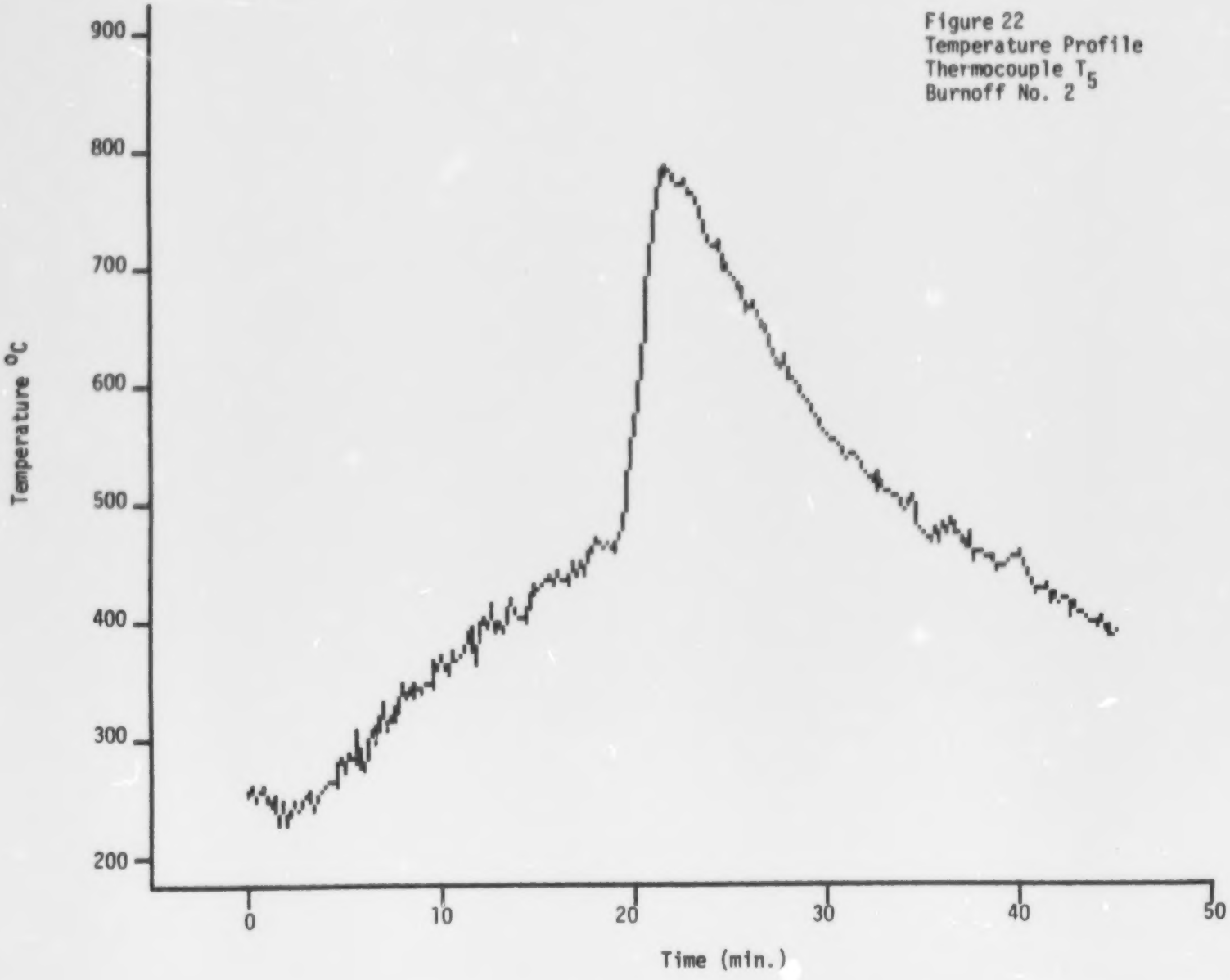


Figure 22
Temperature Profile
Thermocouple T_5
Burnoff No. 2

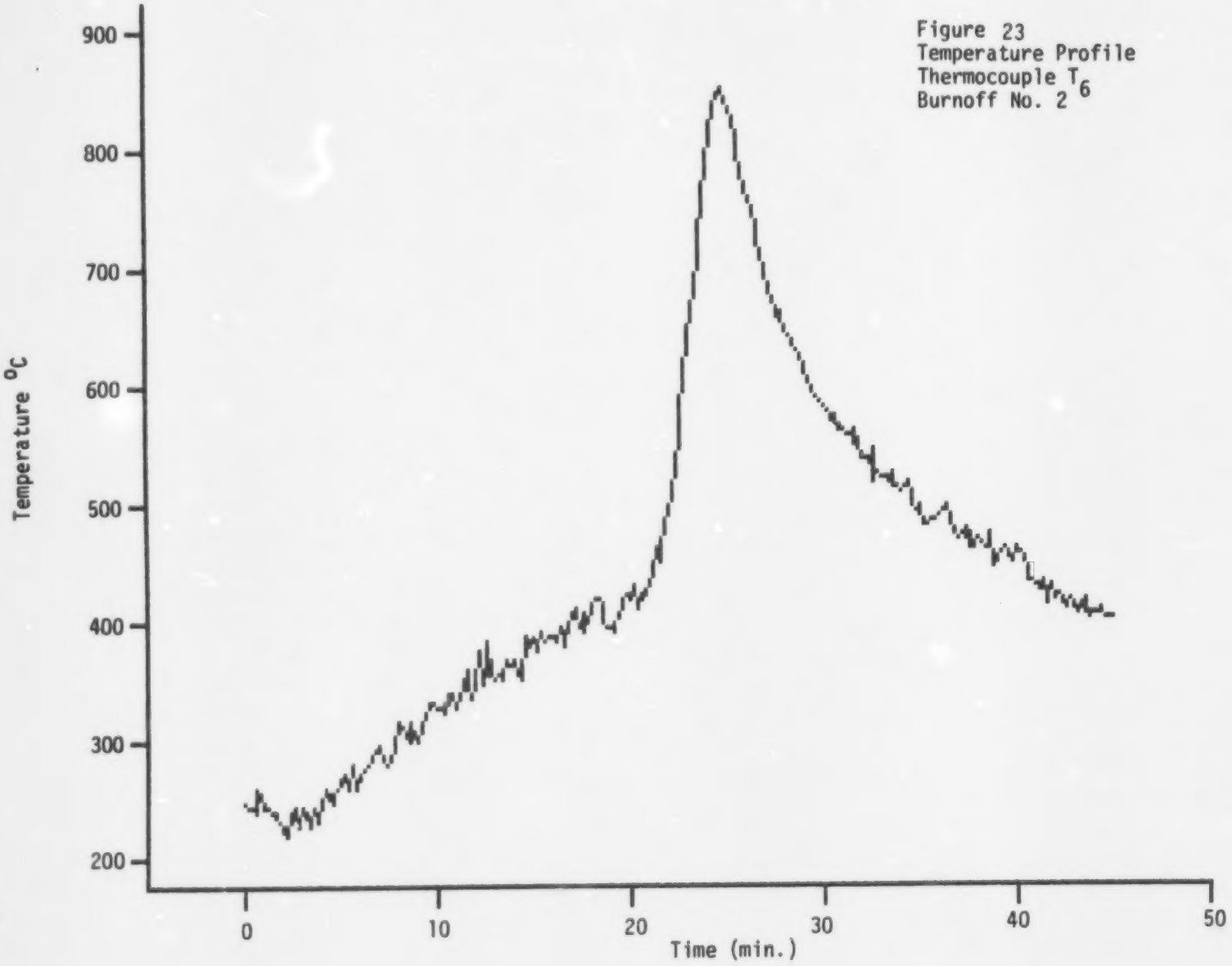


Figure 23
Temperature Profile
Thermocouple T_6
Burnoff No. 2

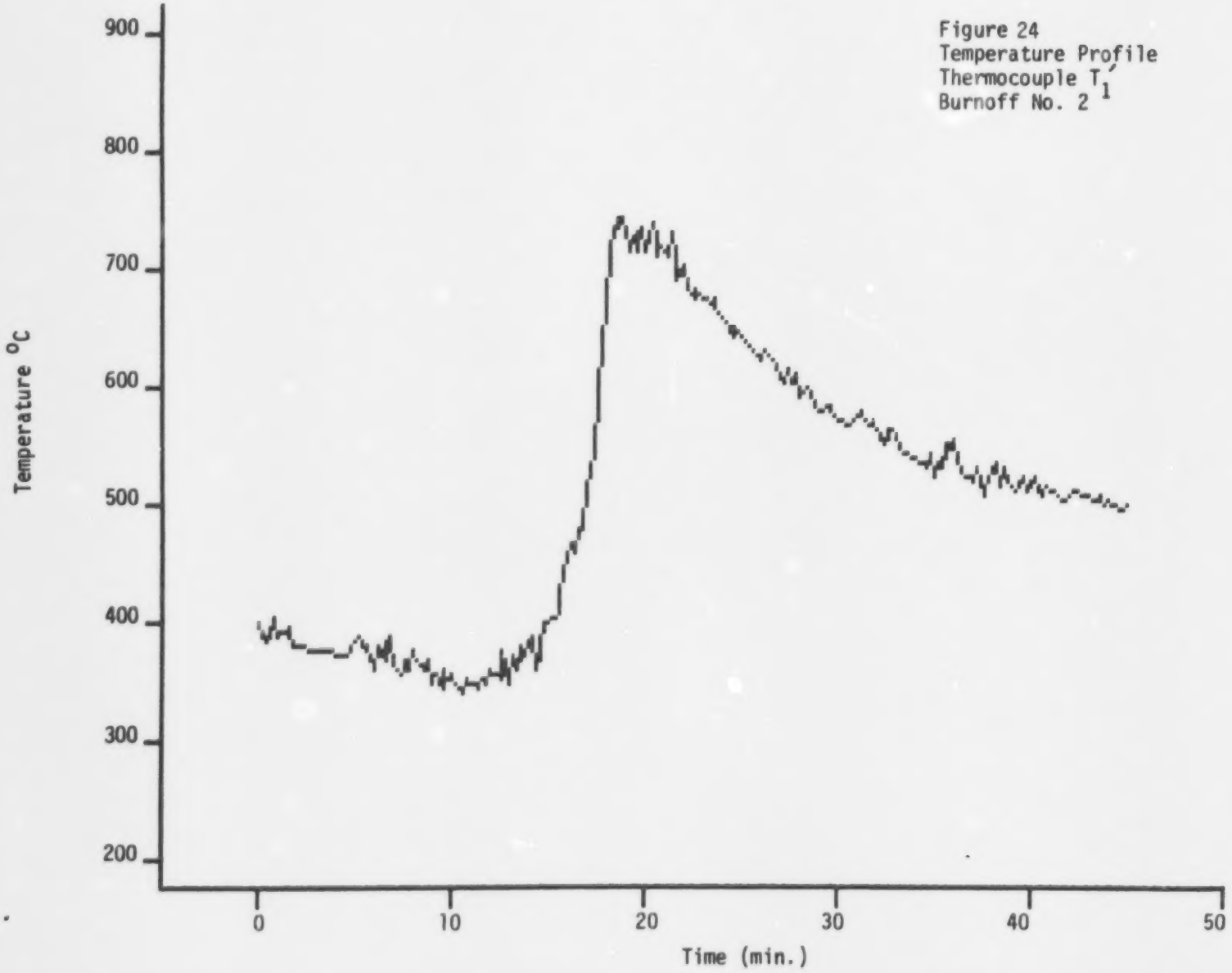


Figure 24
Temperature Profile
Thermocouple T_1'
Burnoff No. 2

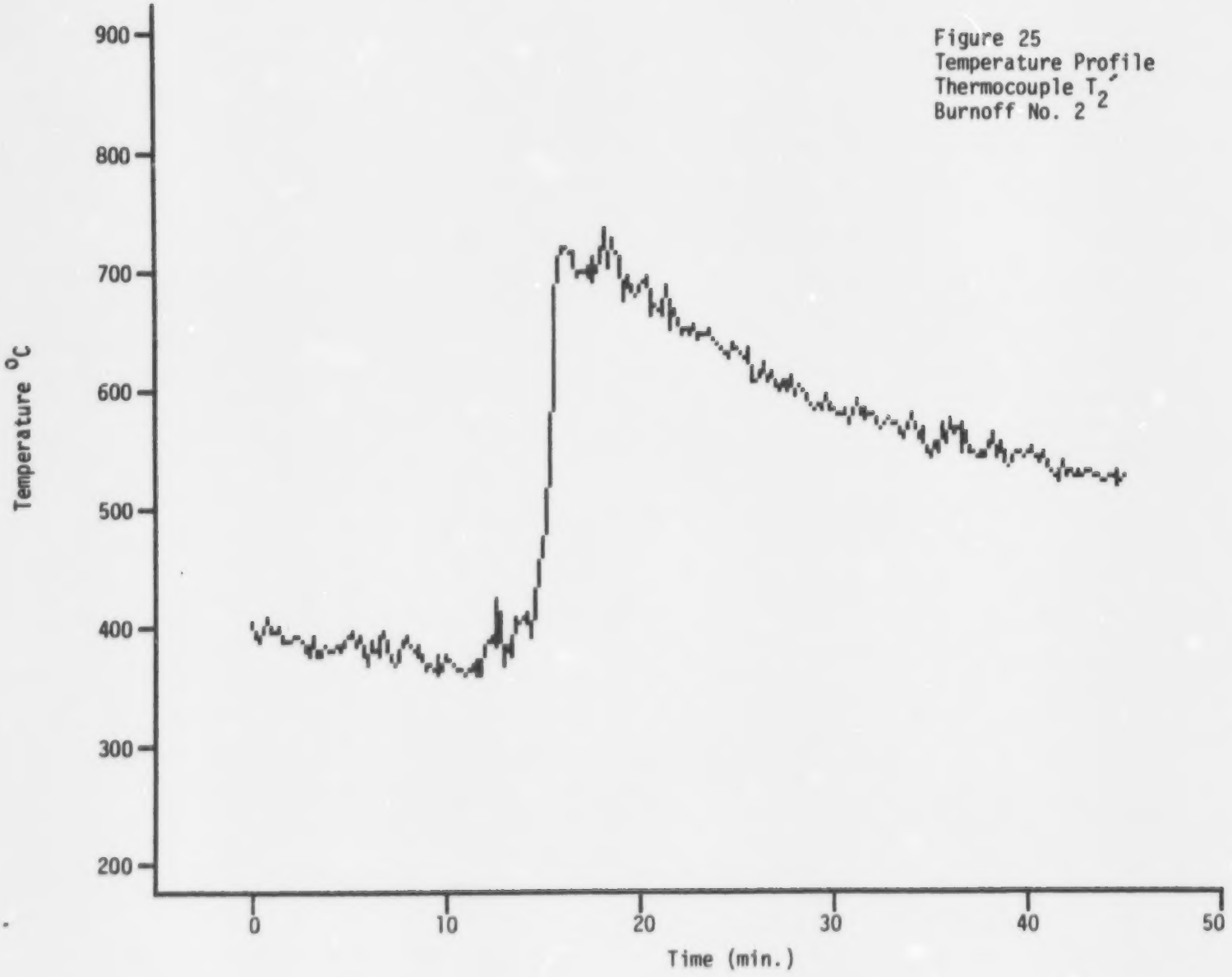


Figure 25
Temperature Profile
Thermocouple T_2'
Burnoff No. 2

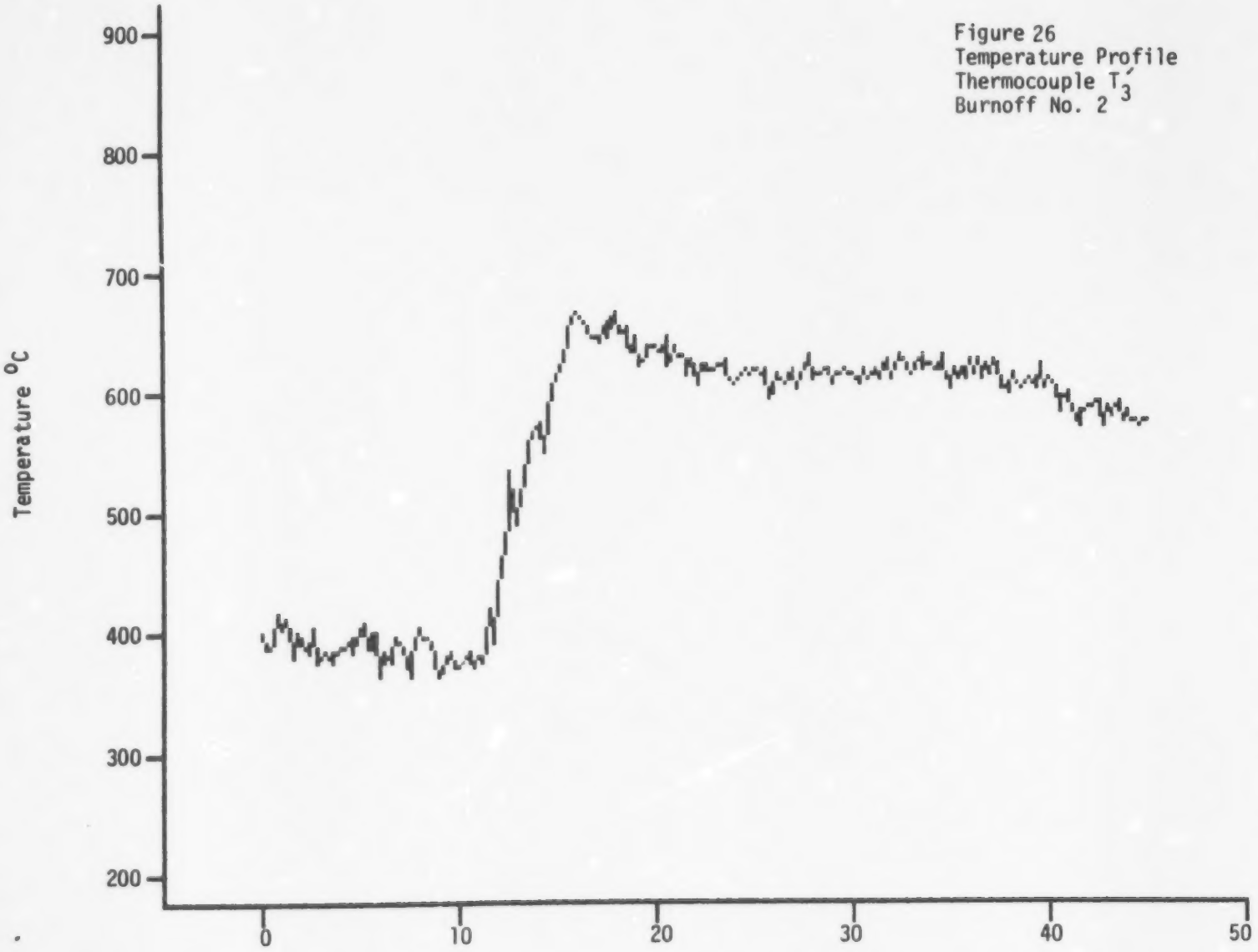


Figure 26
Temperature Profile
Thermocouple T_3'
Burnoff No. 2

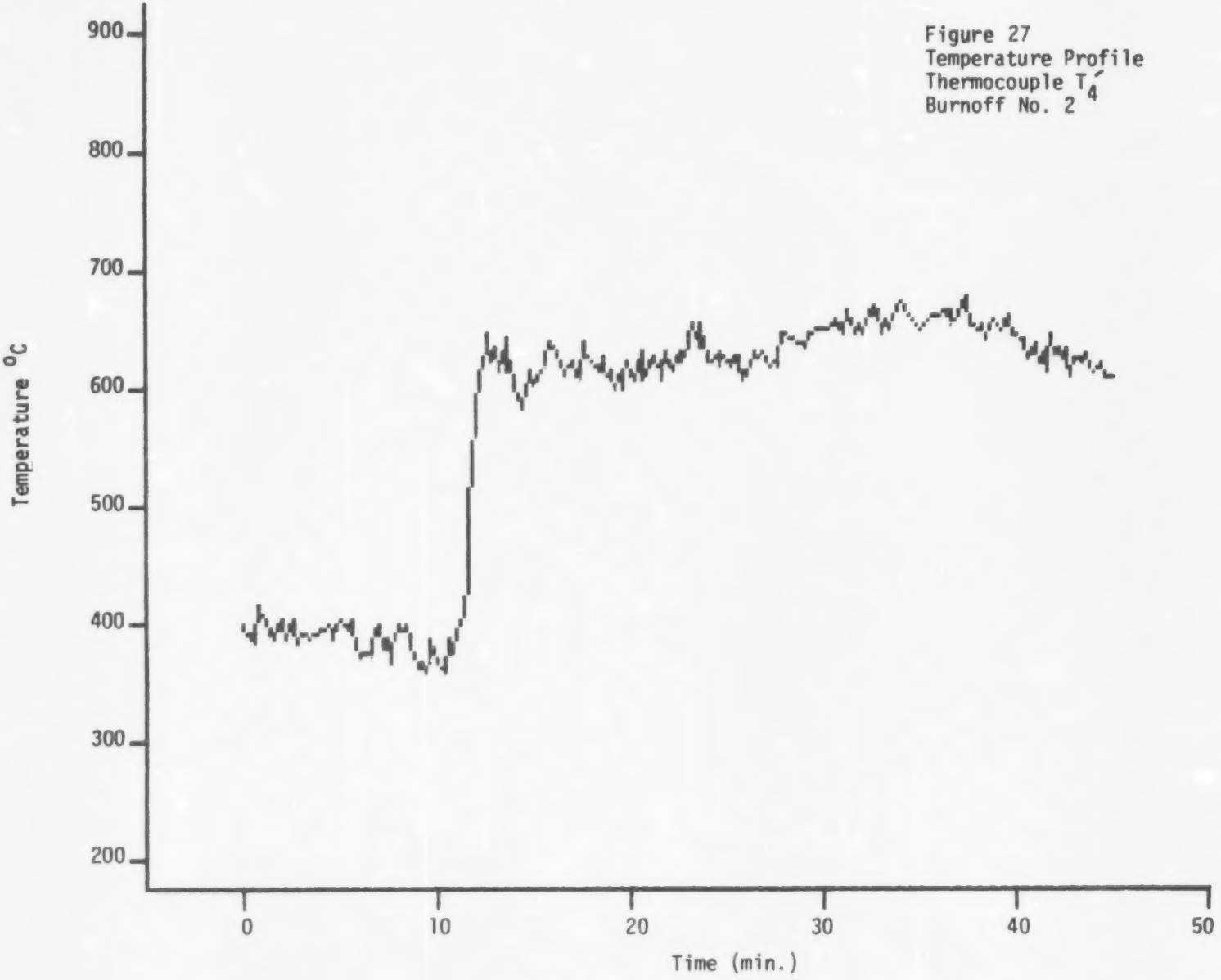


Figure 27
Temperature Profile
Thermocouple T_4'
Burnoff No. 2

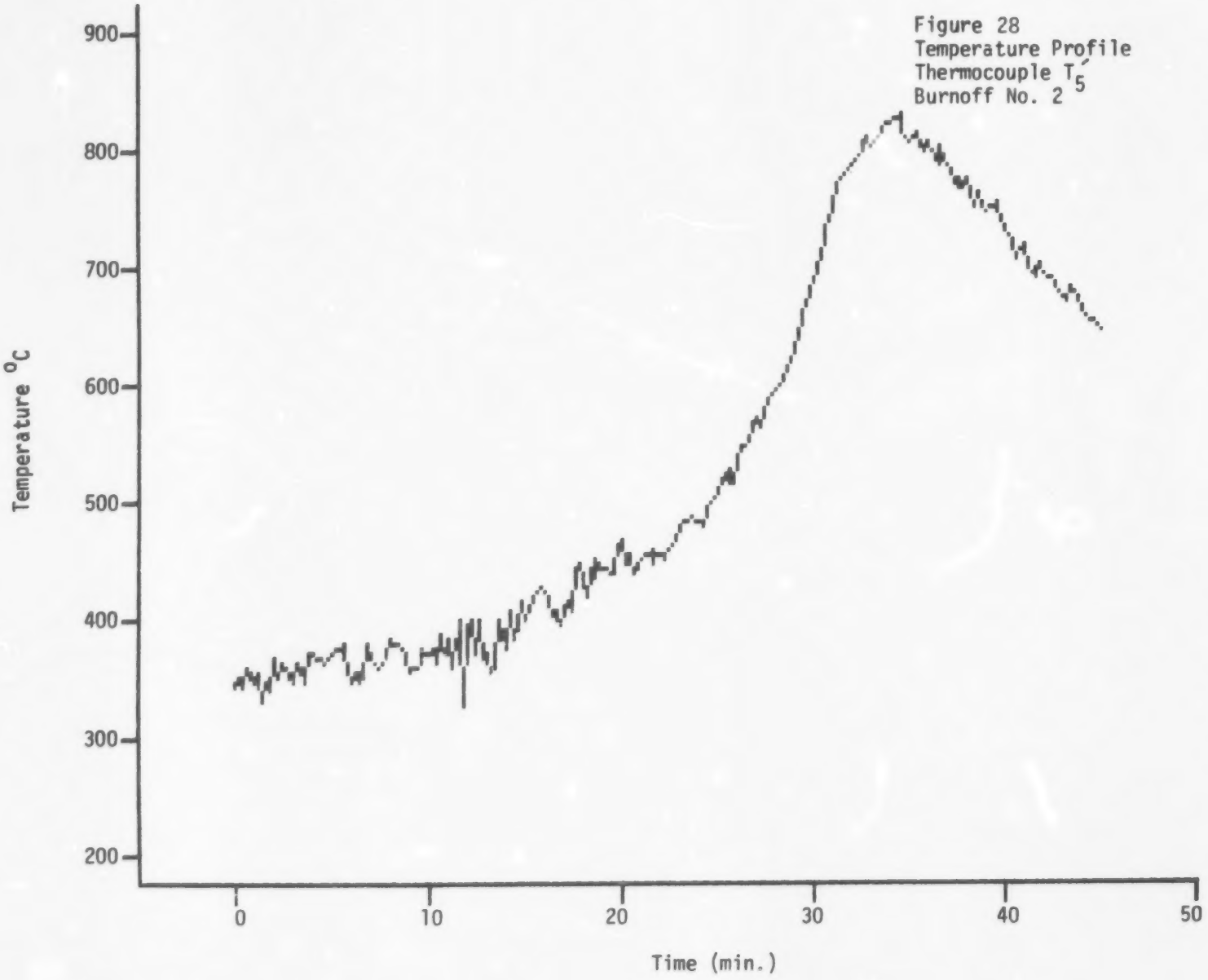


Figure 28
Temperature Profile
Thermocouple T_5'
Burnoff No. 2

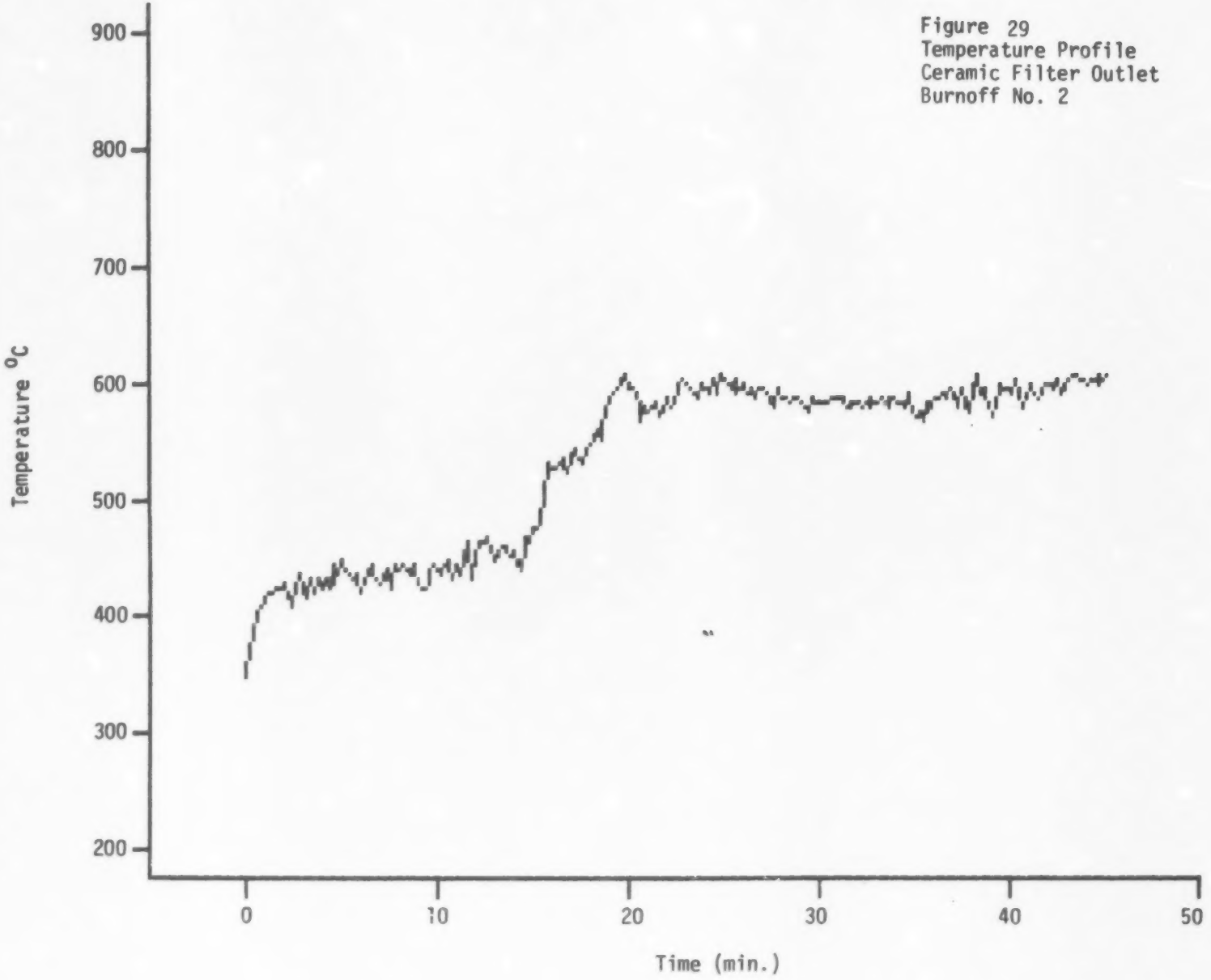


Figure 29
Temperature Profile
Ceramic Filter Outlet
Burnoff No. 2

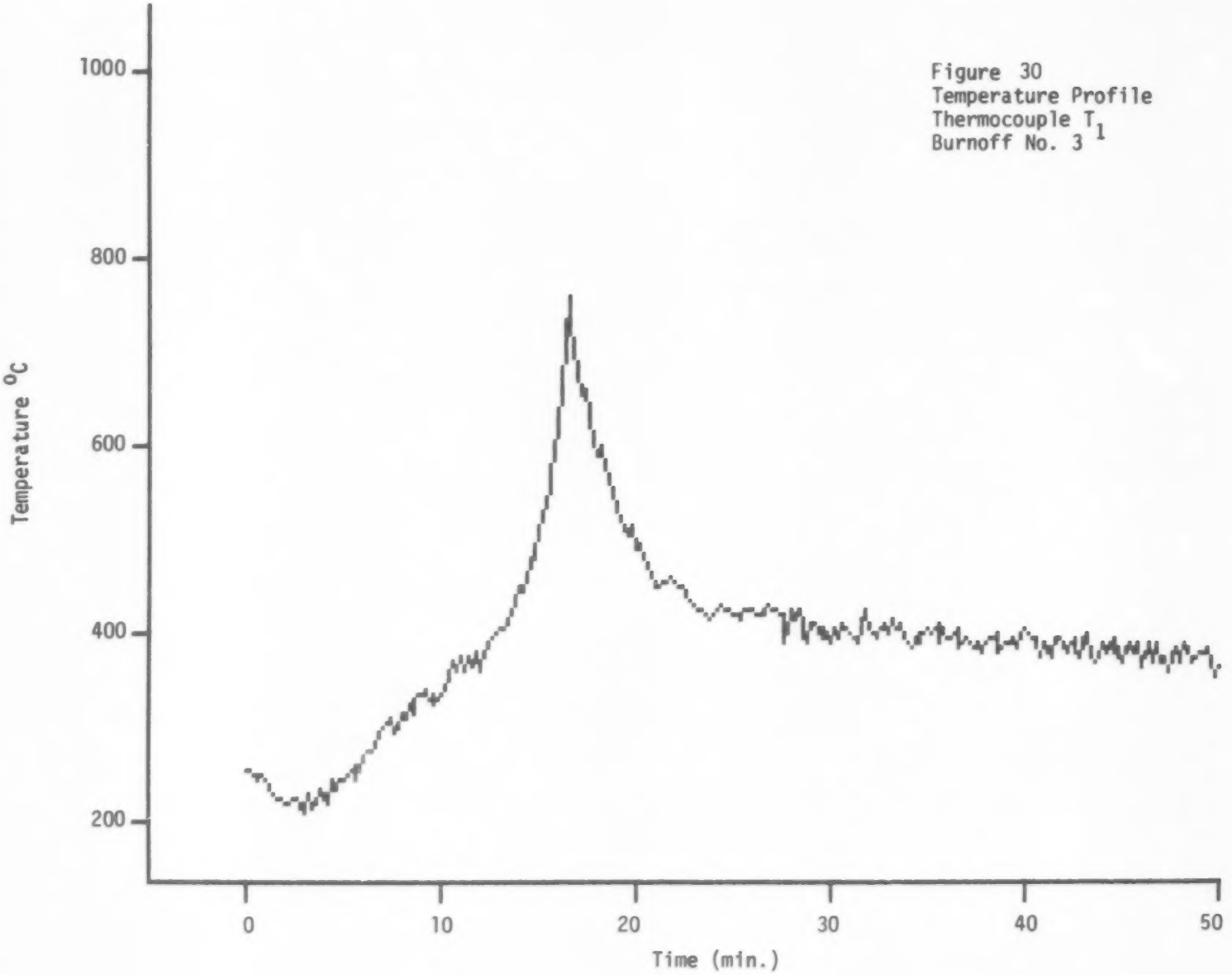


Figure 30
Temperature Profile
Thermocouple T_1
Burnoff No. 3

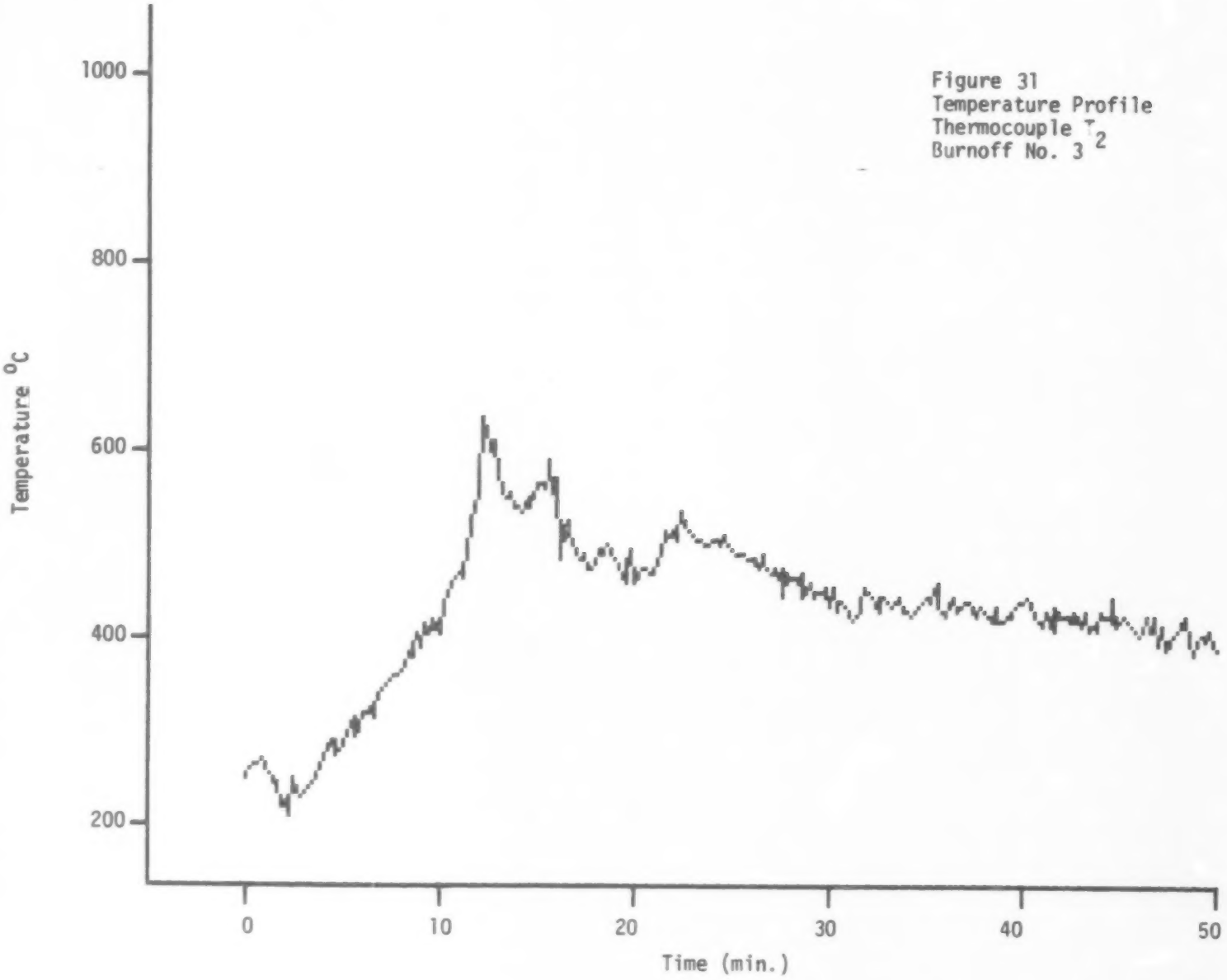


Figure 31
Temperature Profile
Thermocouple T_2
Burnoff No. 3

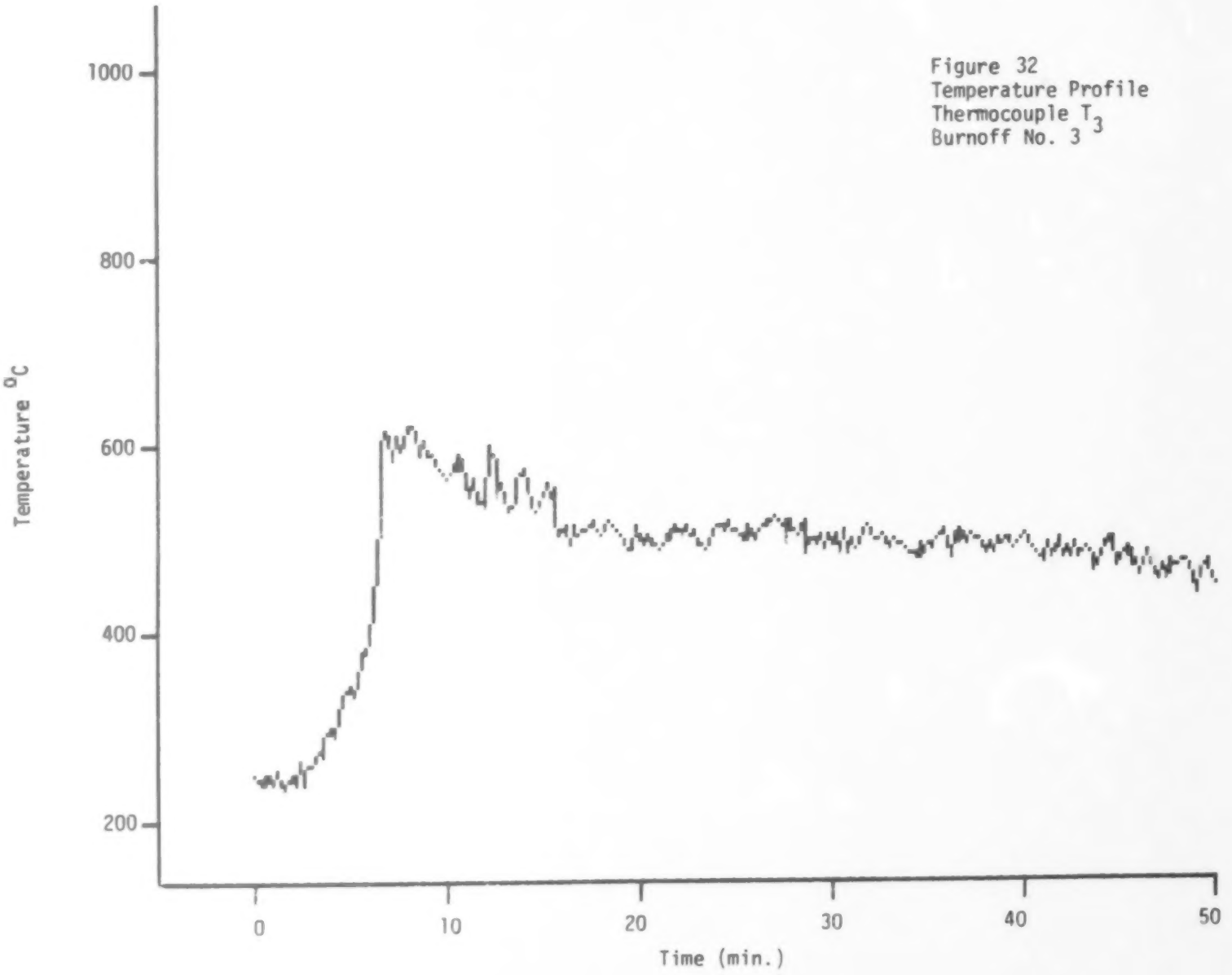


Figure 32
Temperature Profile
Thermocouple T_3
Burnoff No. 3

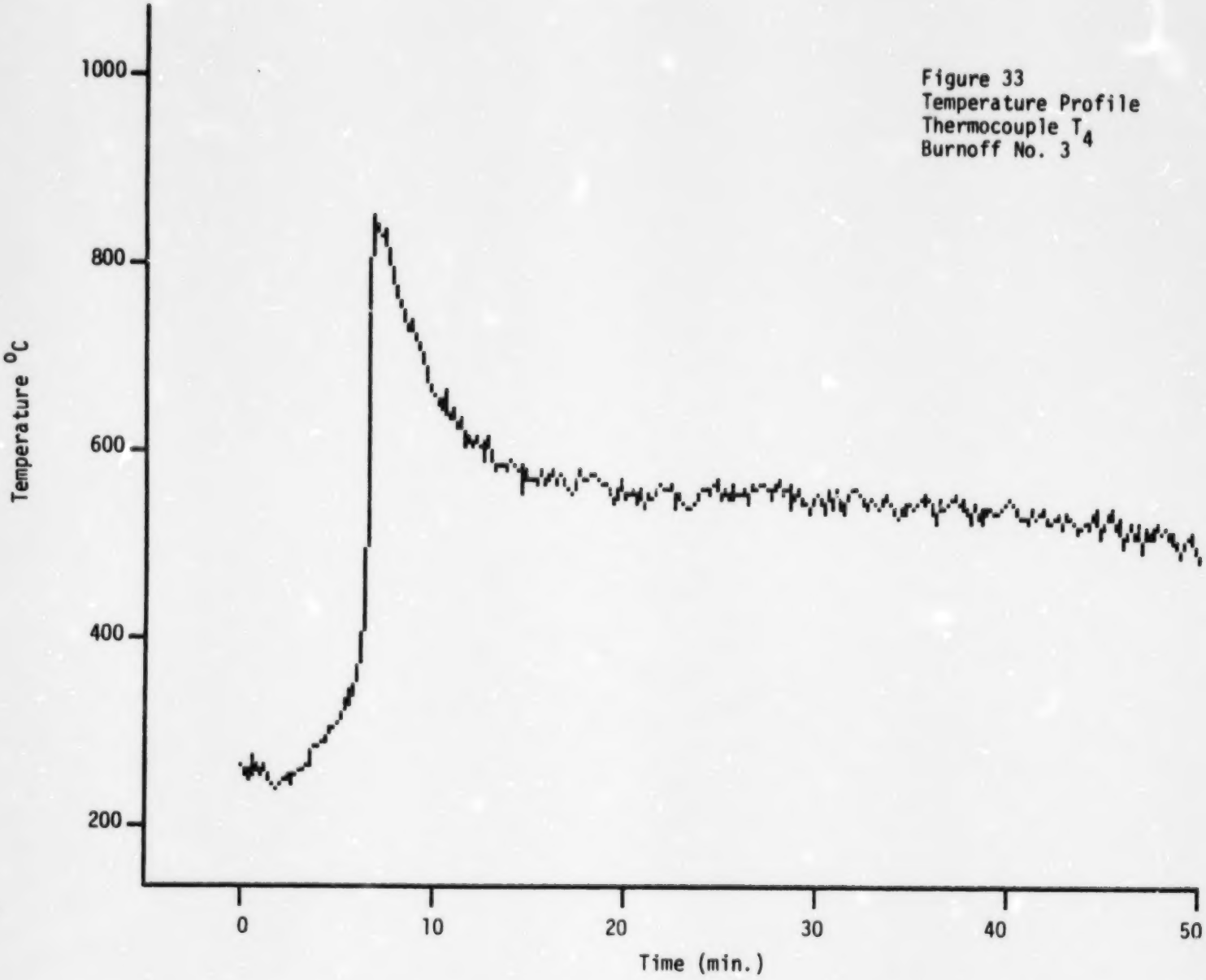


Figure 33
Temperature Profile
Thermocouple T_4
Burnoff No. 3

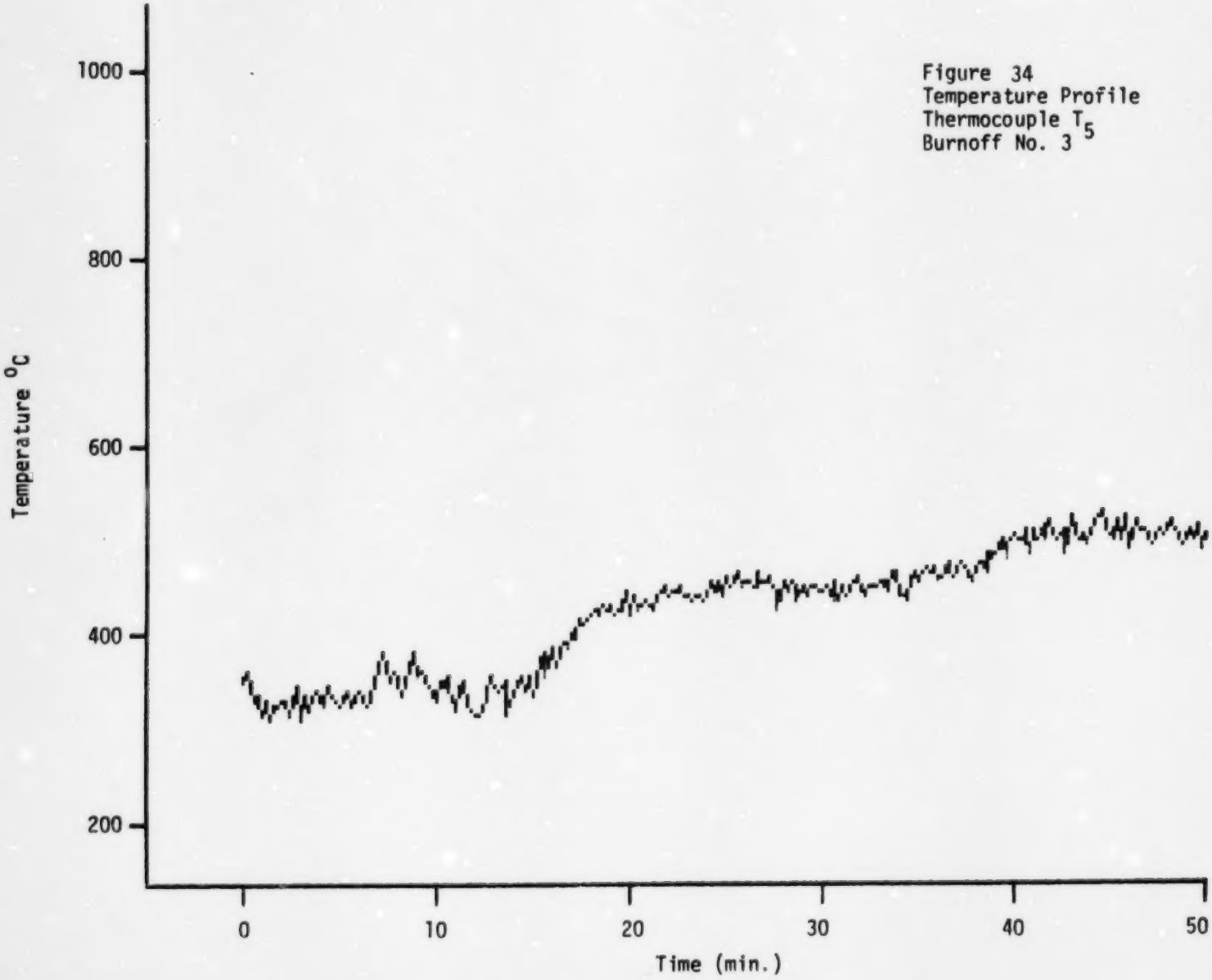


Figure 34
Temperature Profile
Thermocouple T_5
Burnoff No. 3

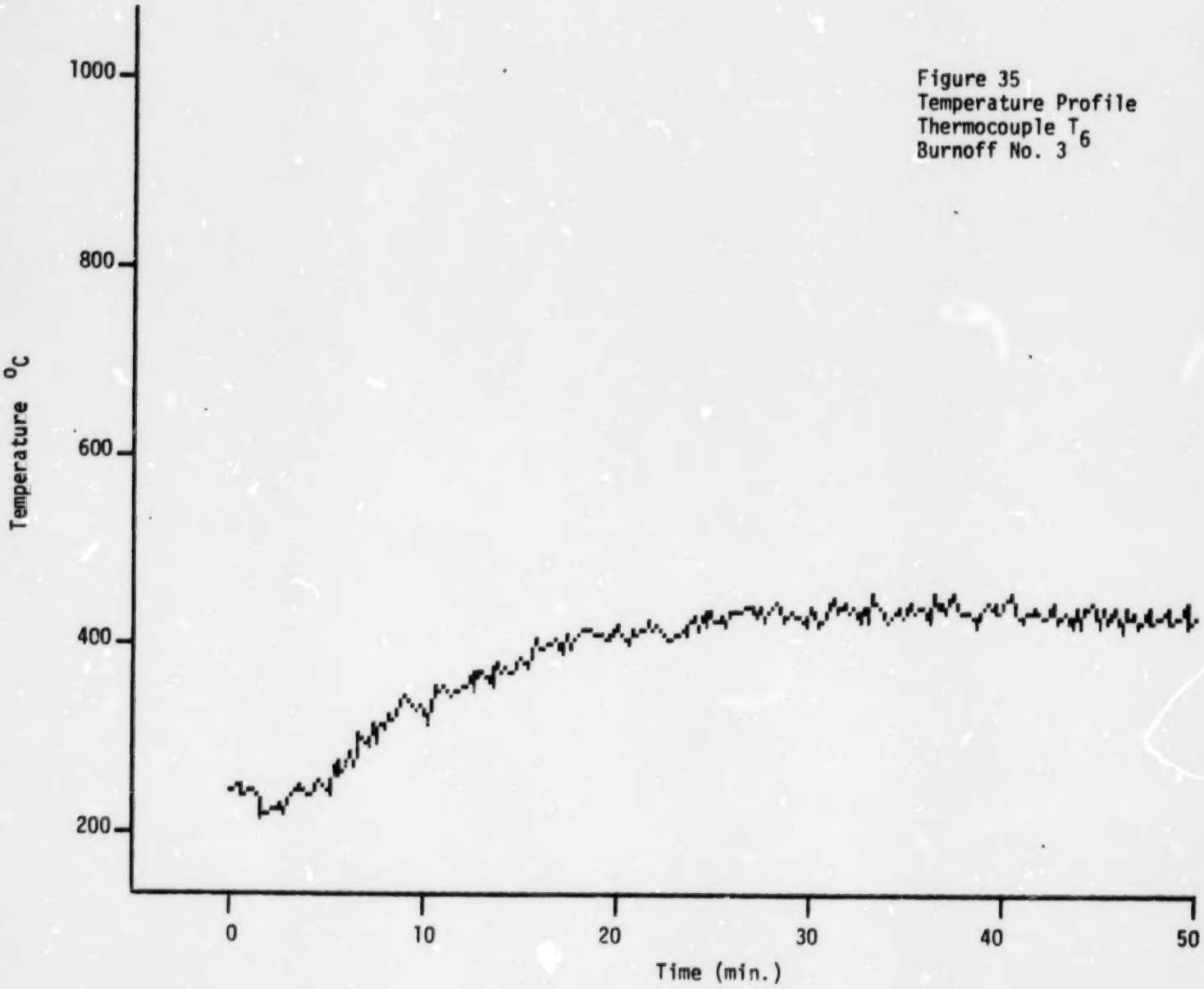


Figure 35
Temperature Profile
Thermocouple T_6
Burnoff No. 3

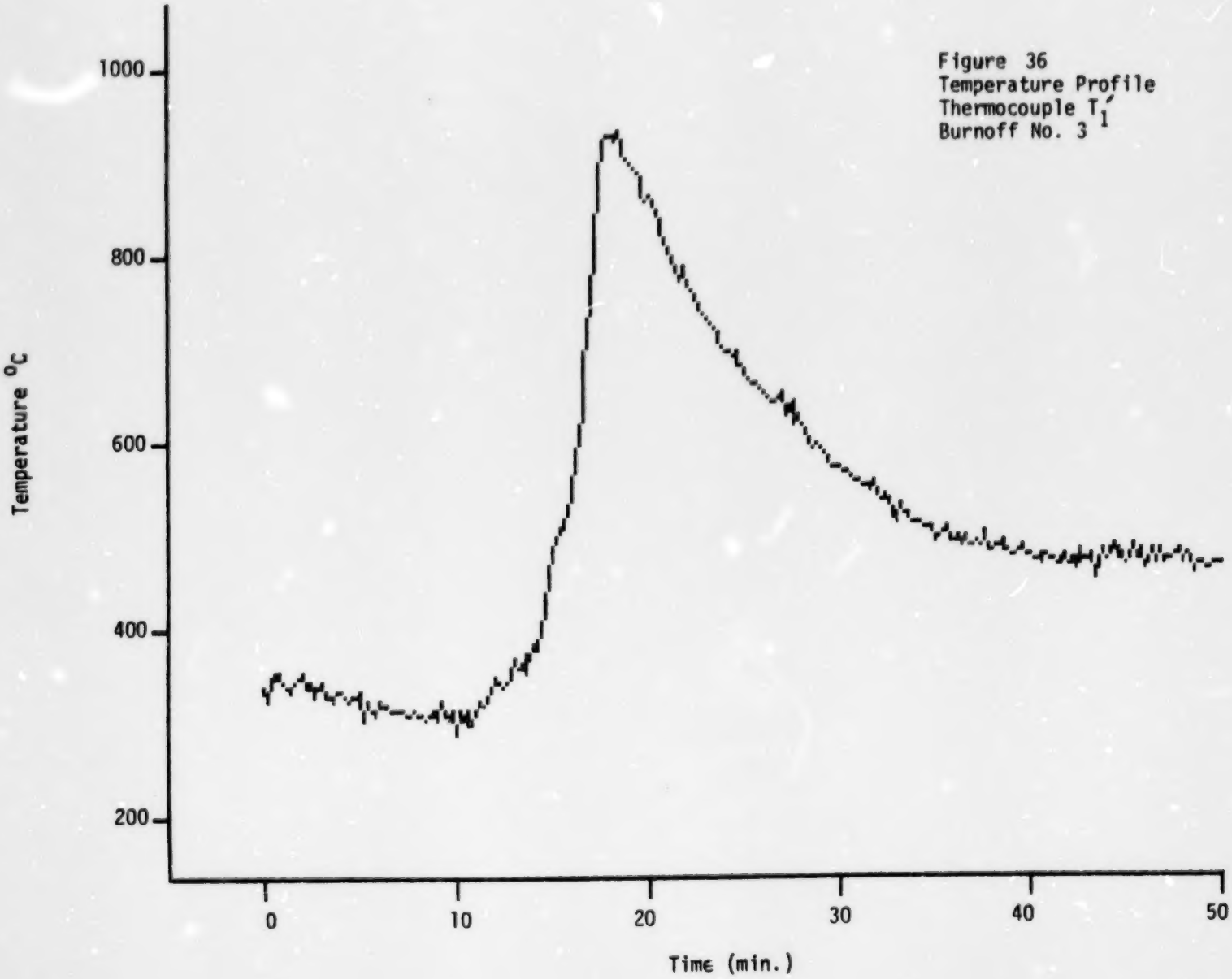


Figure 36
Temperature Profile
Thermocouple T_1'
Burnoff No. 3

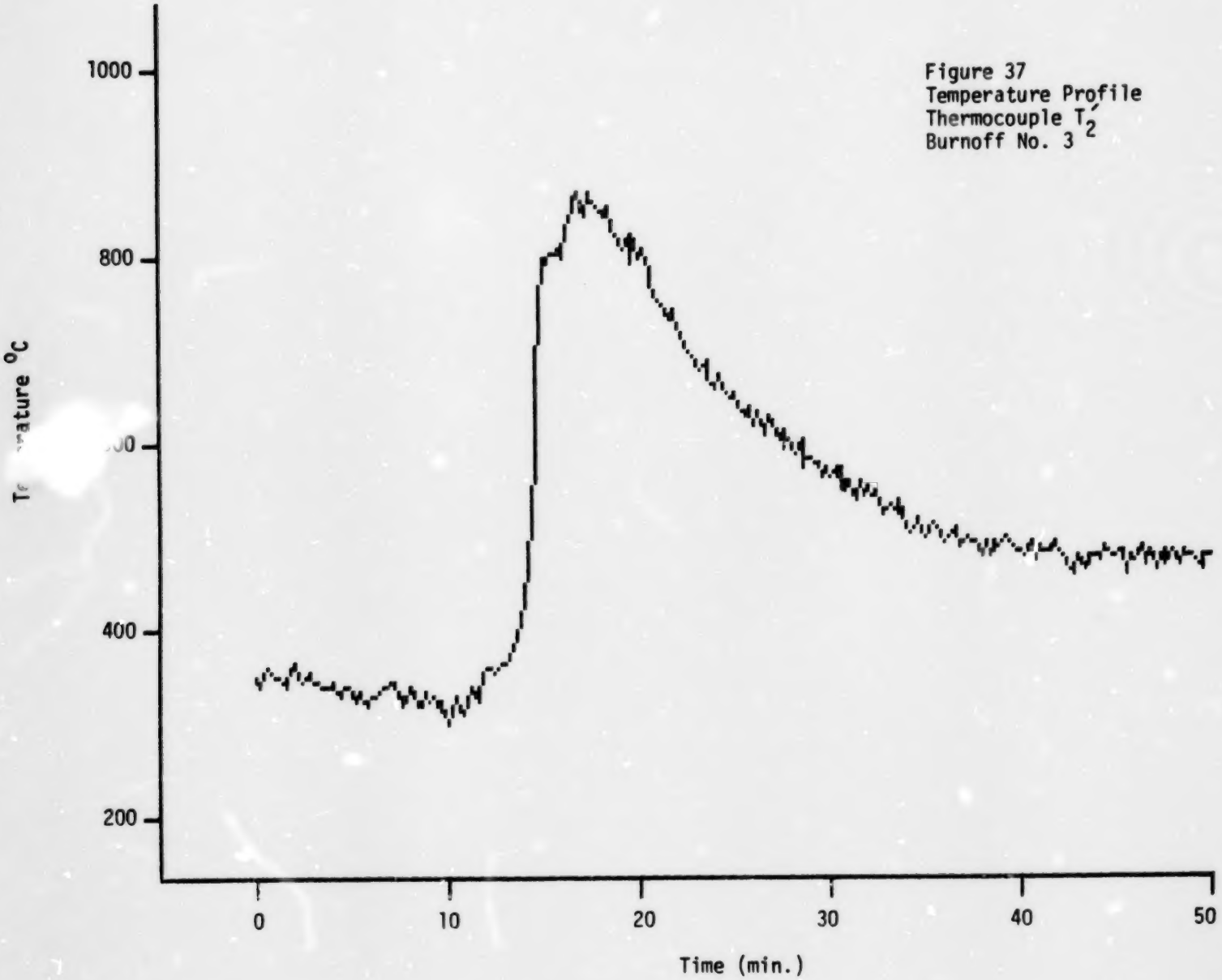


Figure 37
Temperature Profile
Thermocouple T_2'
Burnoff No. 3

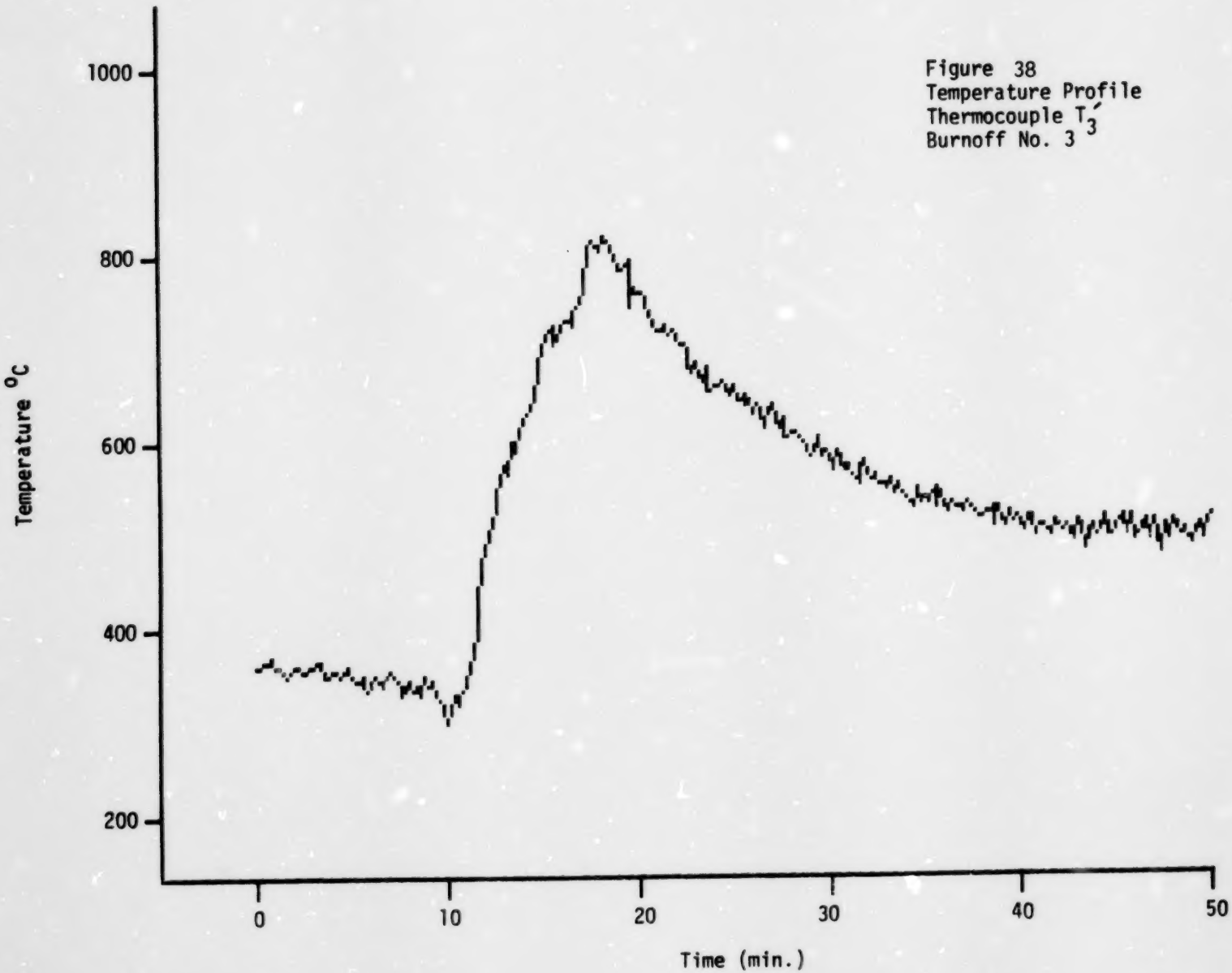


Figure 38
Temperature Profile
Thermocouple T_3'
Burnoff No. 3

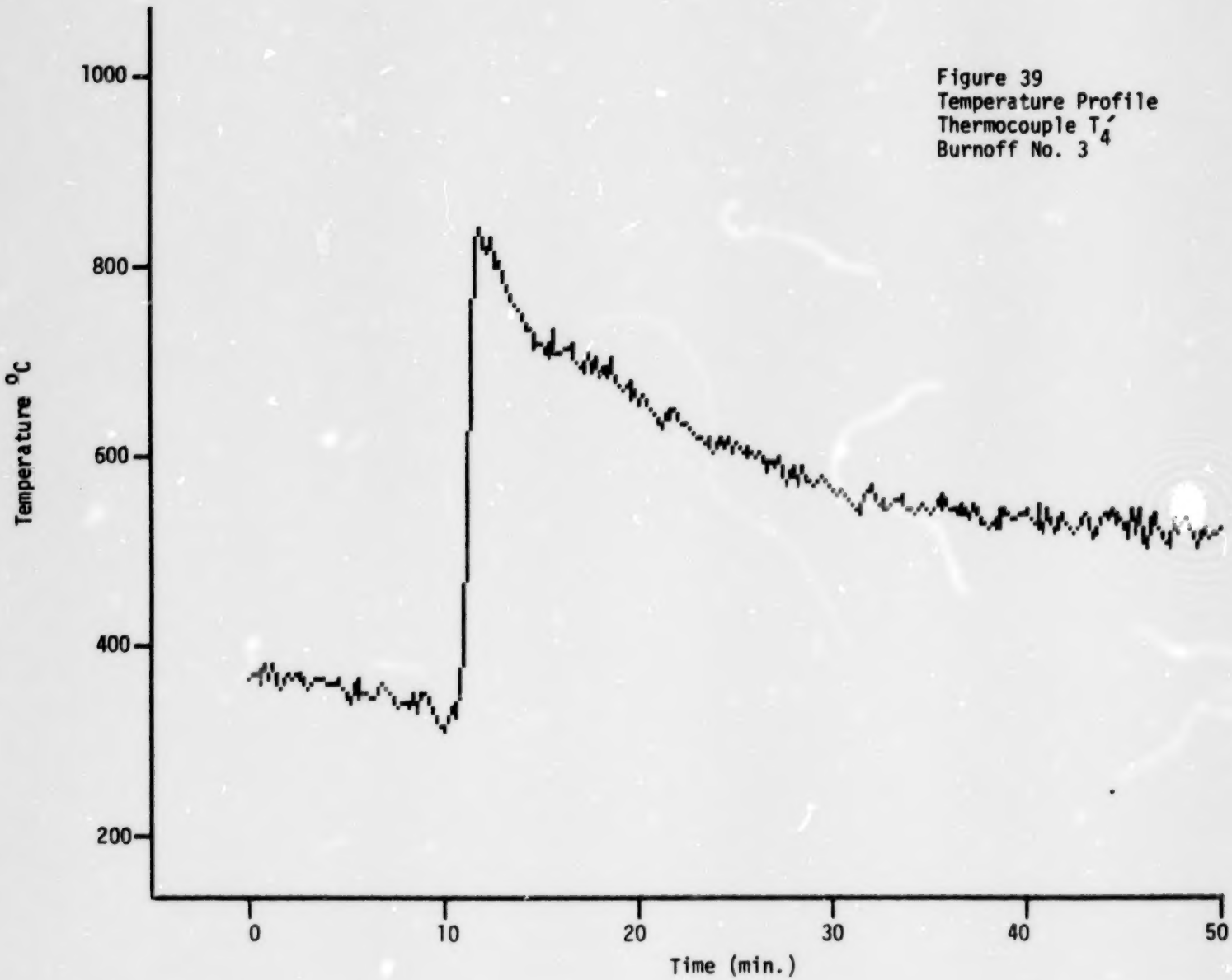


Figure 39
Temperature Profile
Thermocouple T_4'
Burnoff No. 3

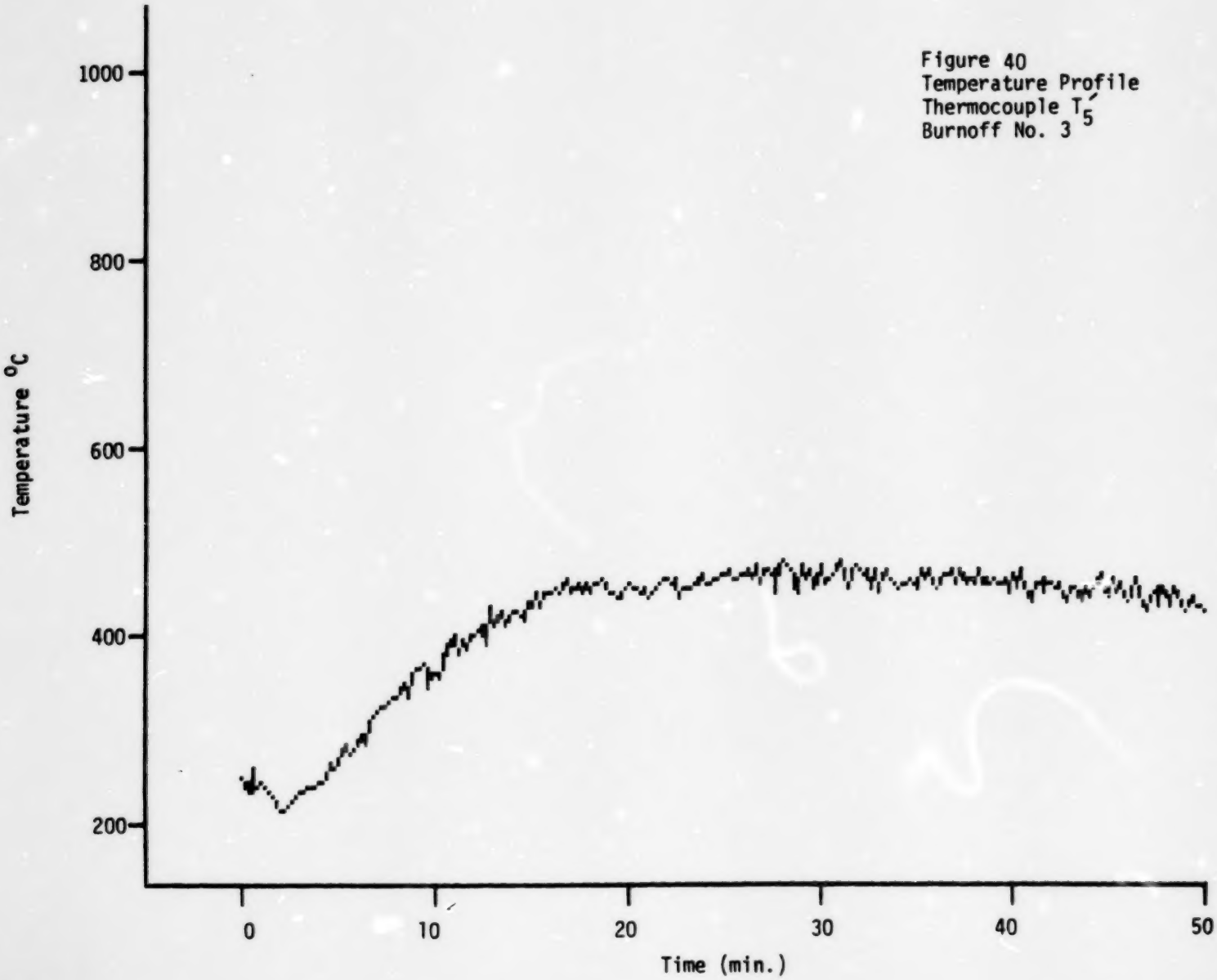


Figure 40
Temperature Profile
Thermocouple T_5'
Burnoff No. 3

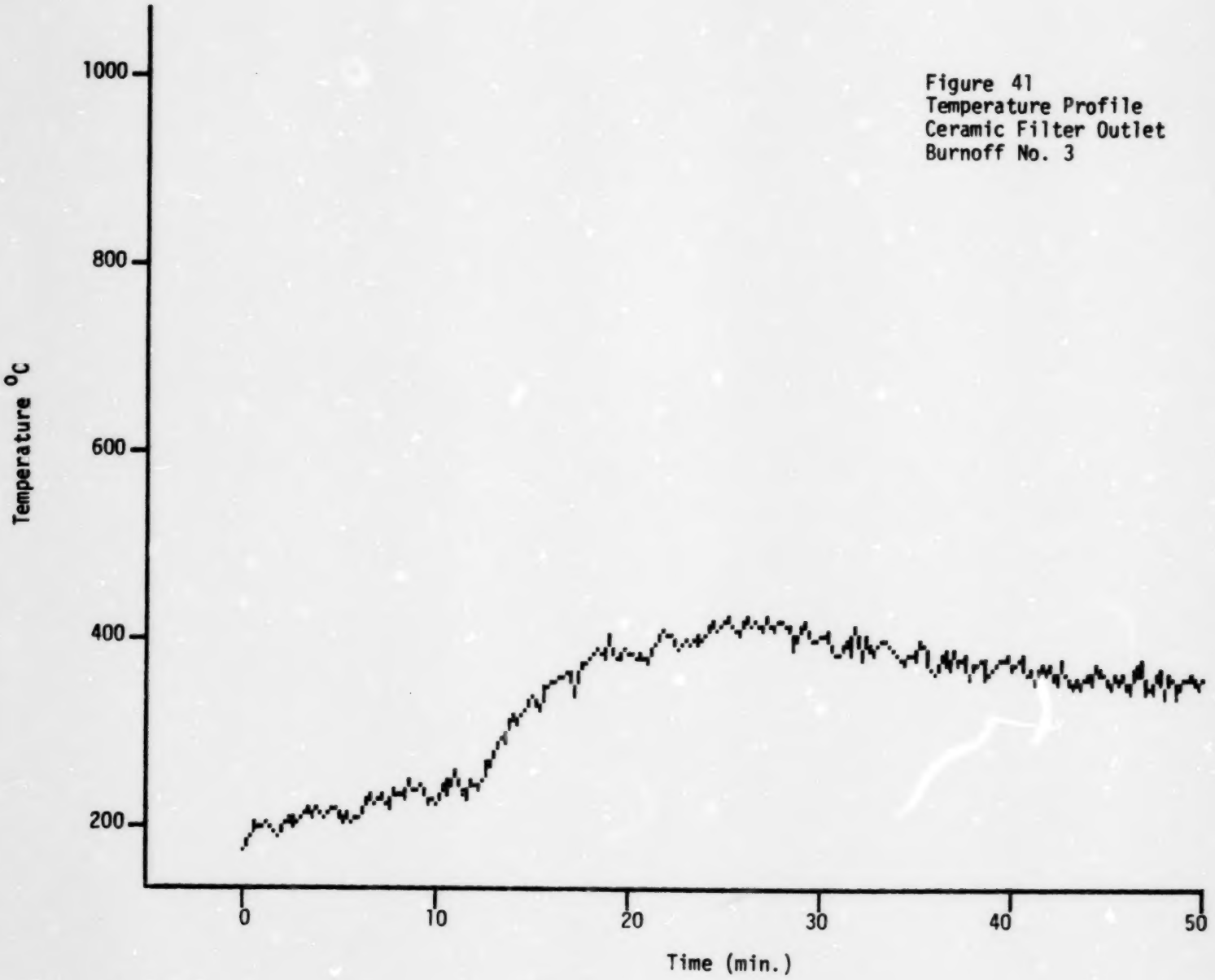


Figure 41
Temperature Profile
Ceramic Filter Outlet
Burnoff No. 3

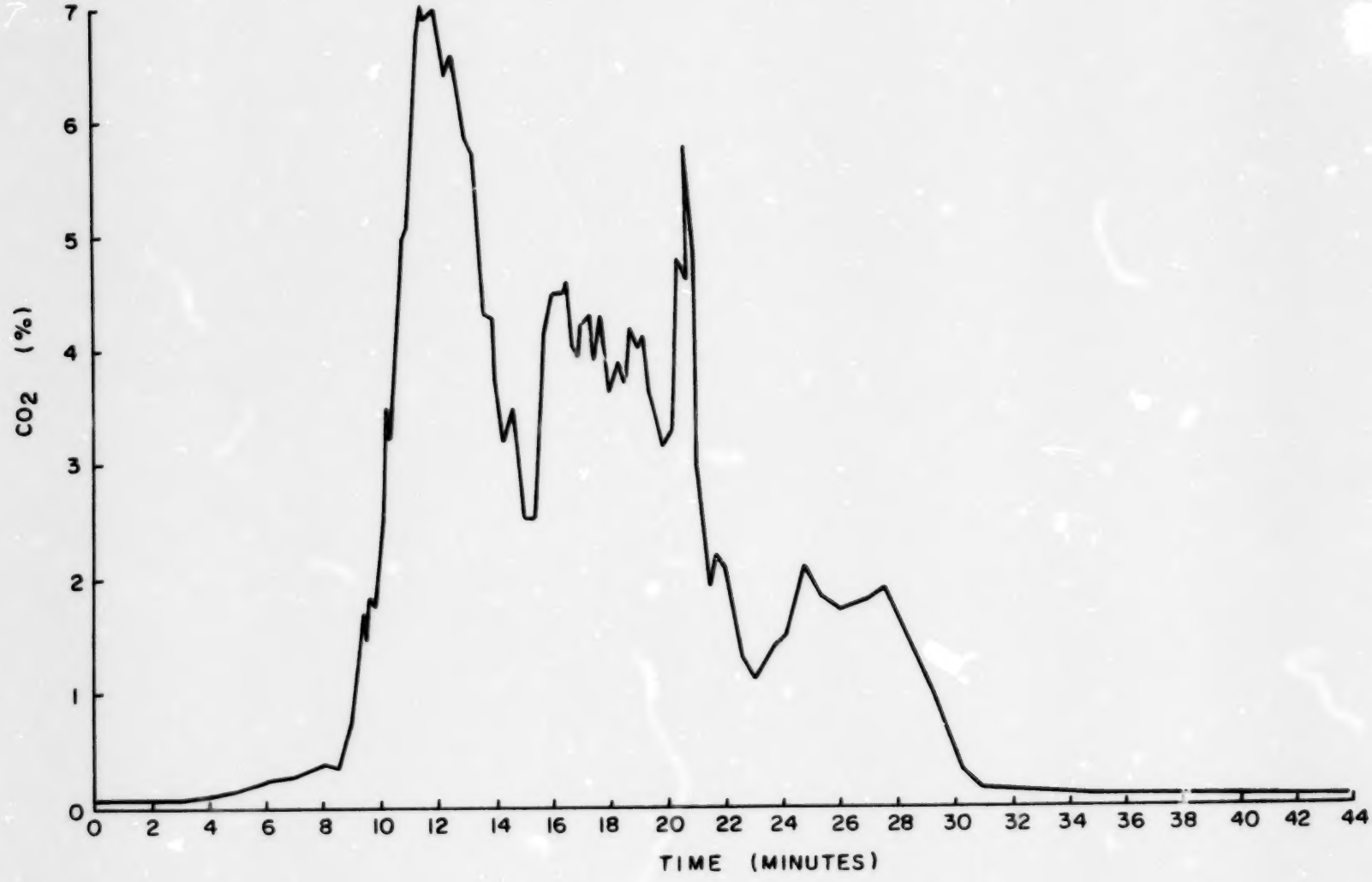


FIGURE 42 CARBON DIOXIDE CONCENTRATION vs TIME - BURNOFF No. 1.

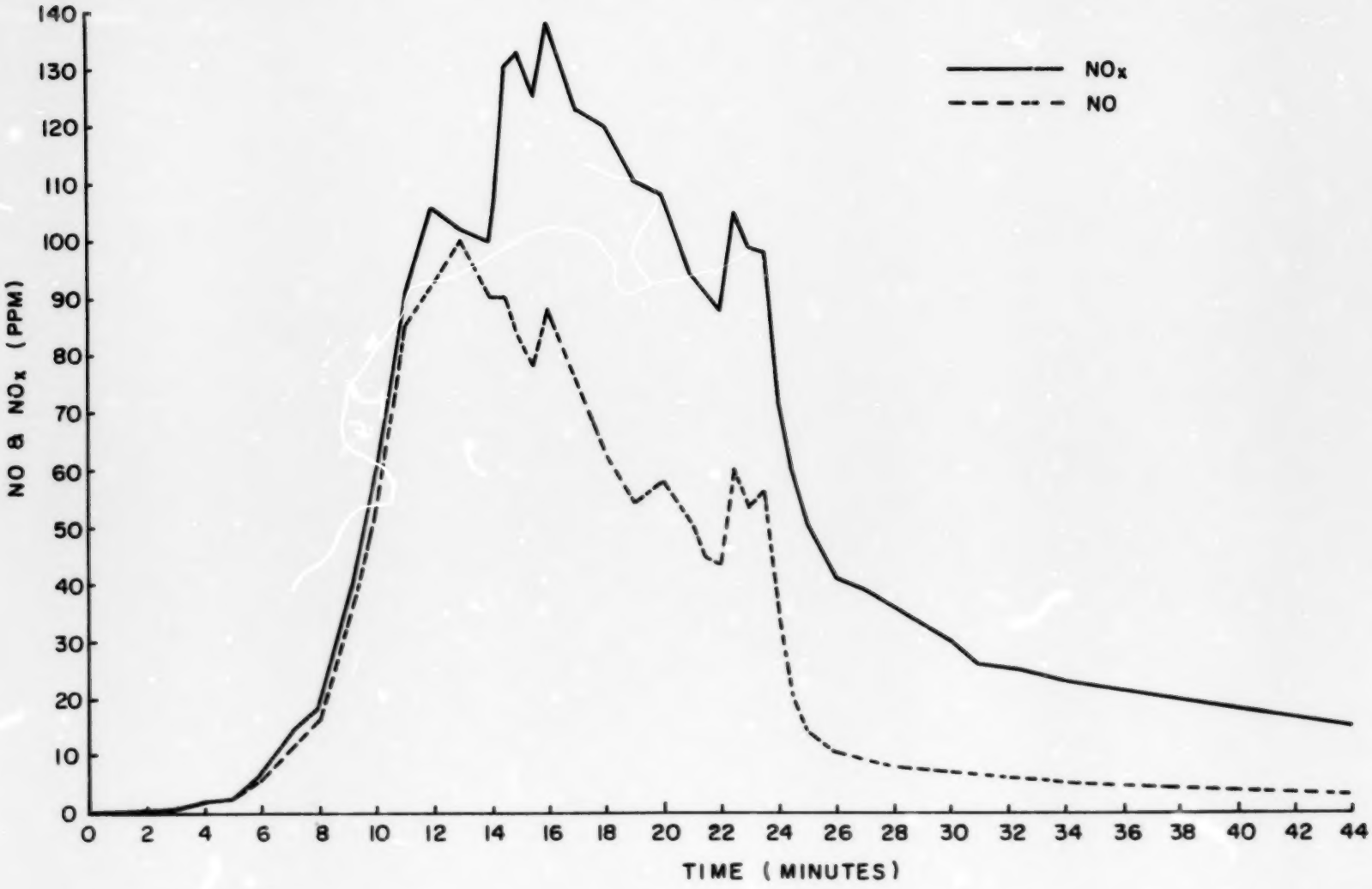


FIGURE 43 CONCENTRATIONS OF NO & NO_x vs TIME - BURNOFF No.1

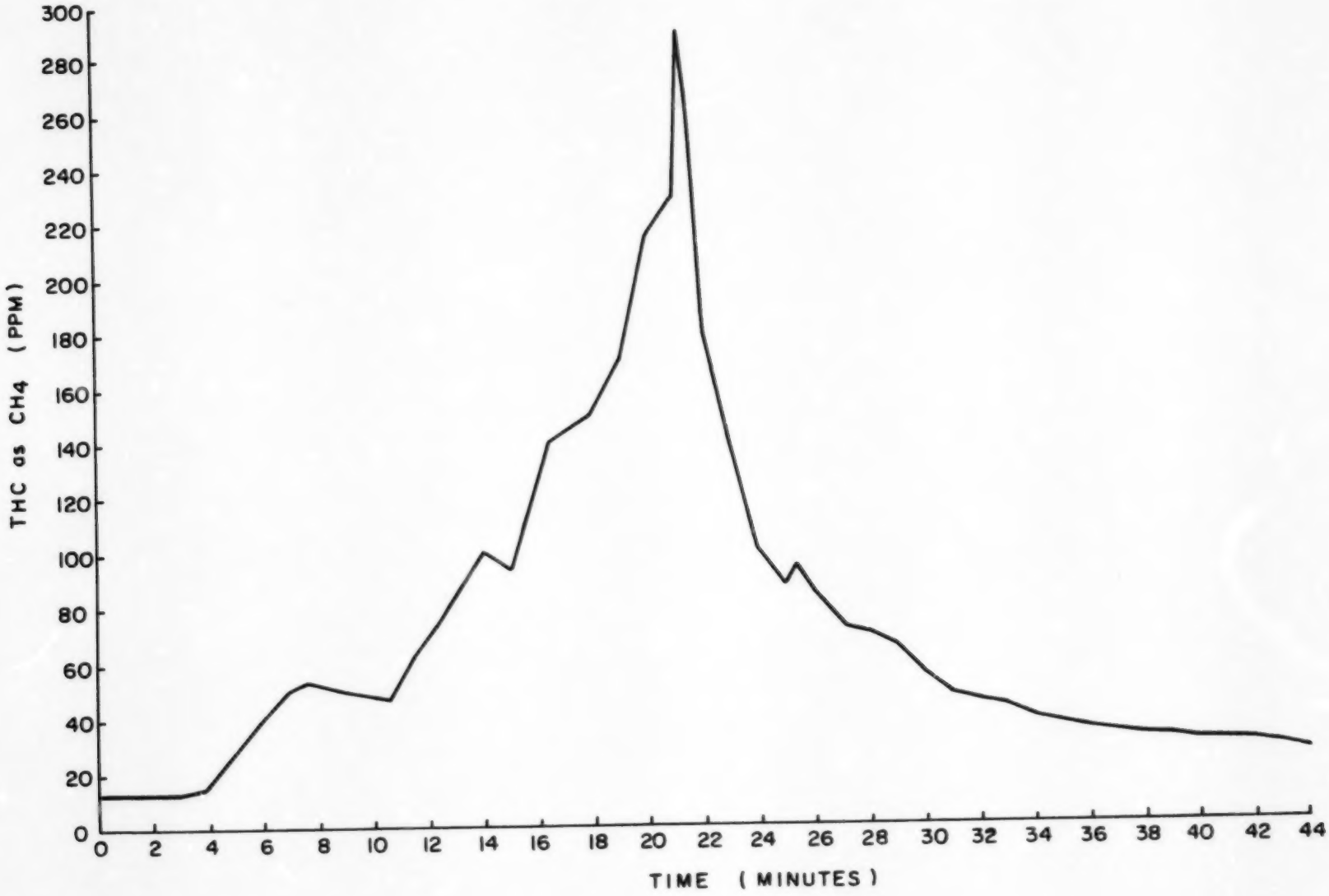


FIGURE 44 TOTAL HYDROCARBON CONCENTRATION vs TIME - BURNOFF No. 1.

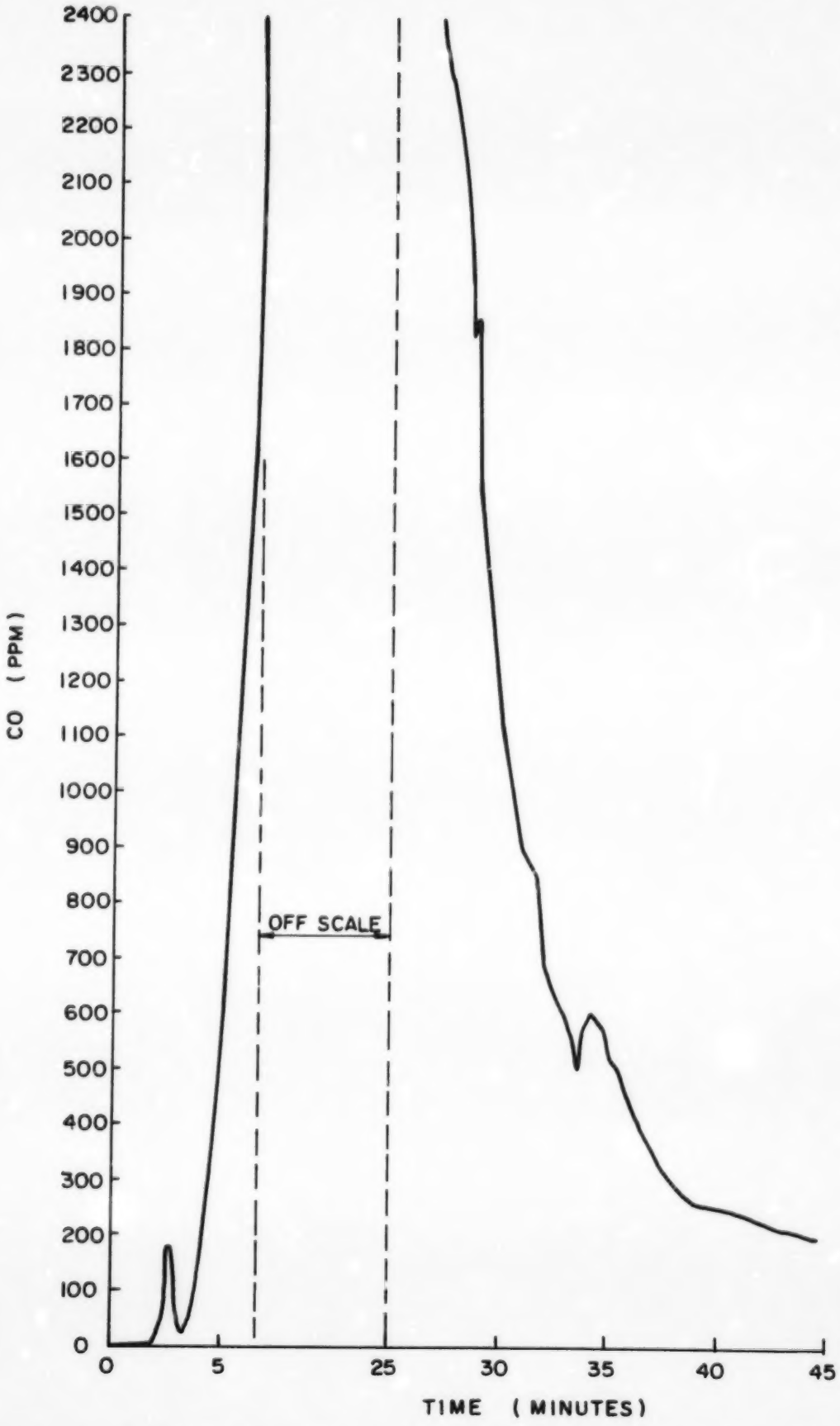


FIGURE 45 CARBON MONOXIDE CONCENTRATION vs TIME - BURNOFF No. 1.

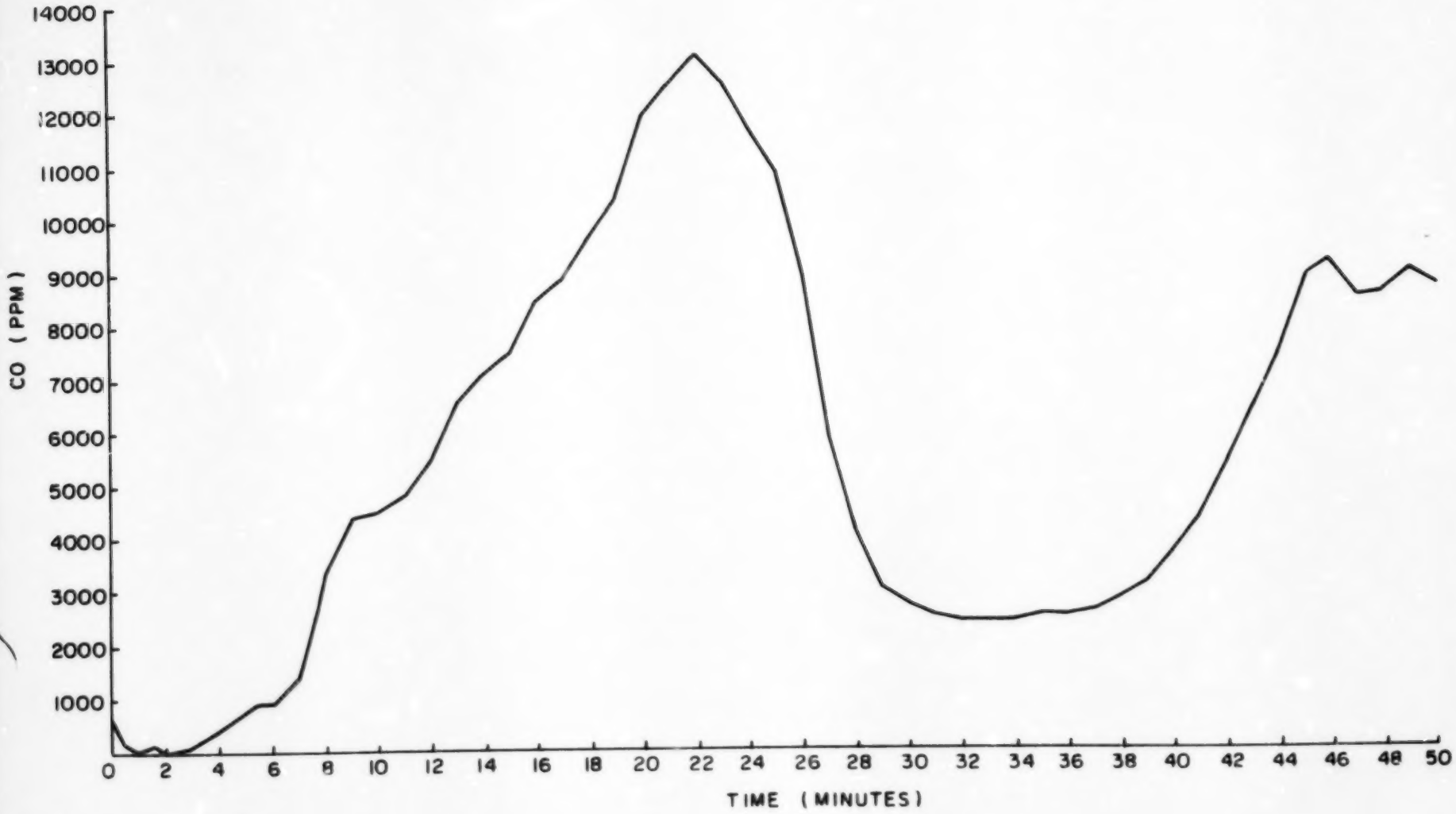


FIGURE 46 CARBON MONOXIDE CONCENTRATION vs TIME - BURNOFF No. 4.

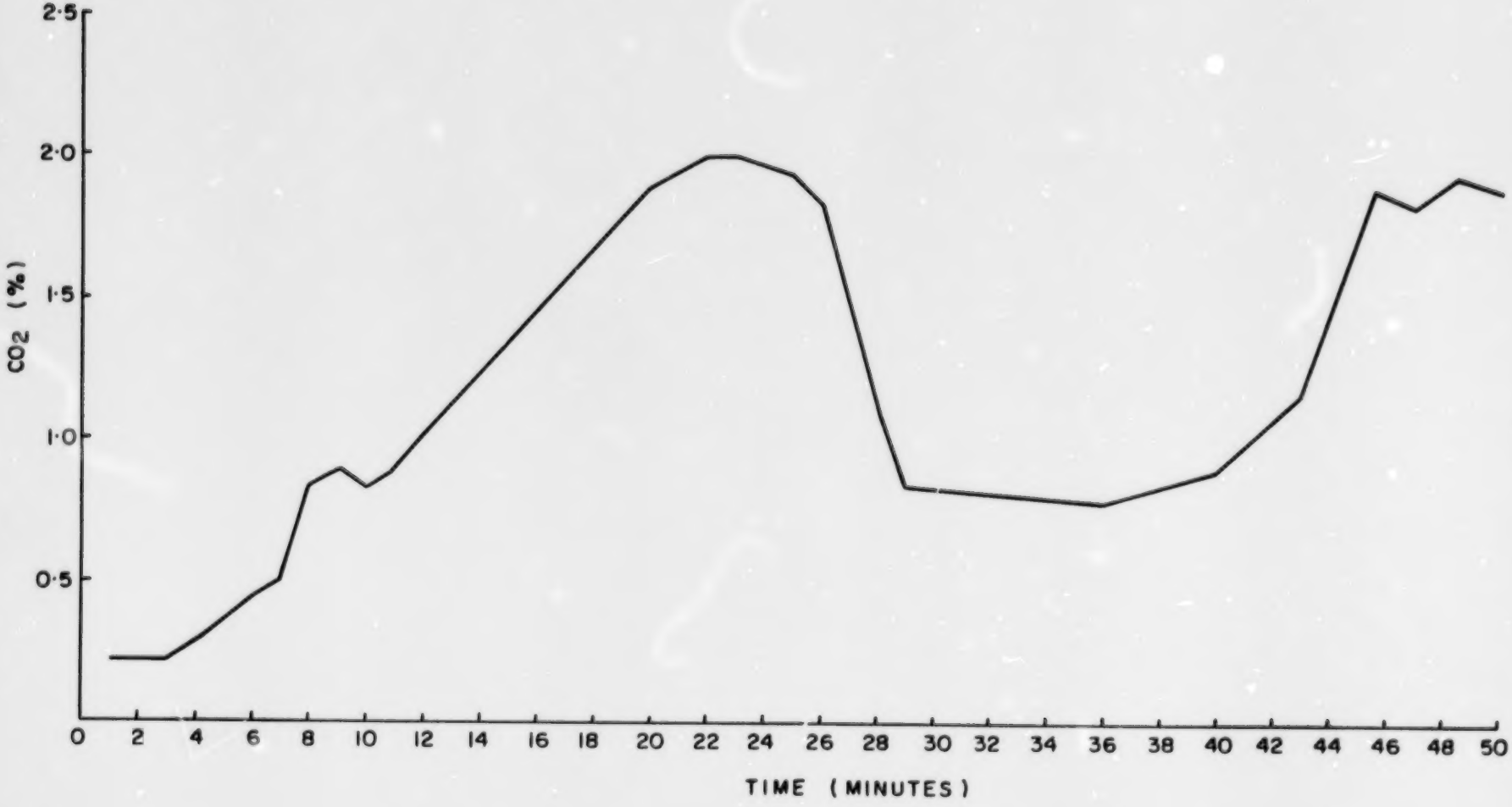


FIGURE 47 CARBON DIOXIDE CONCENTRATION vs TIME - BURNOFF No. 4.

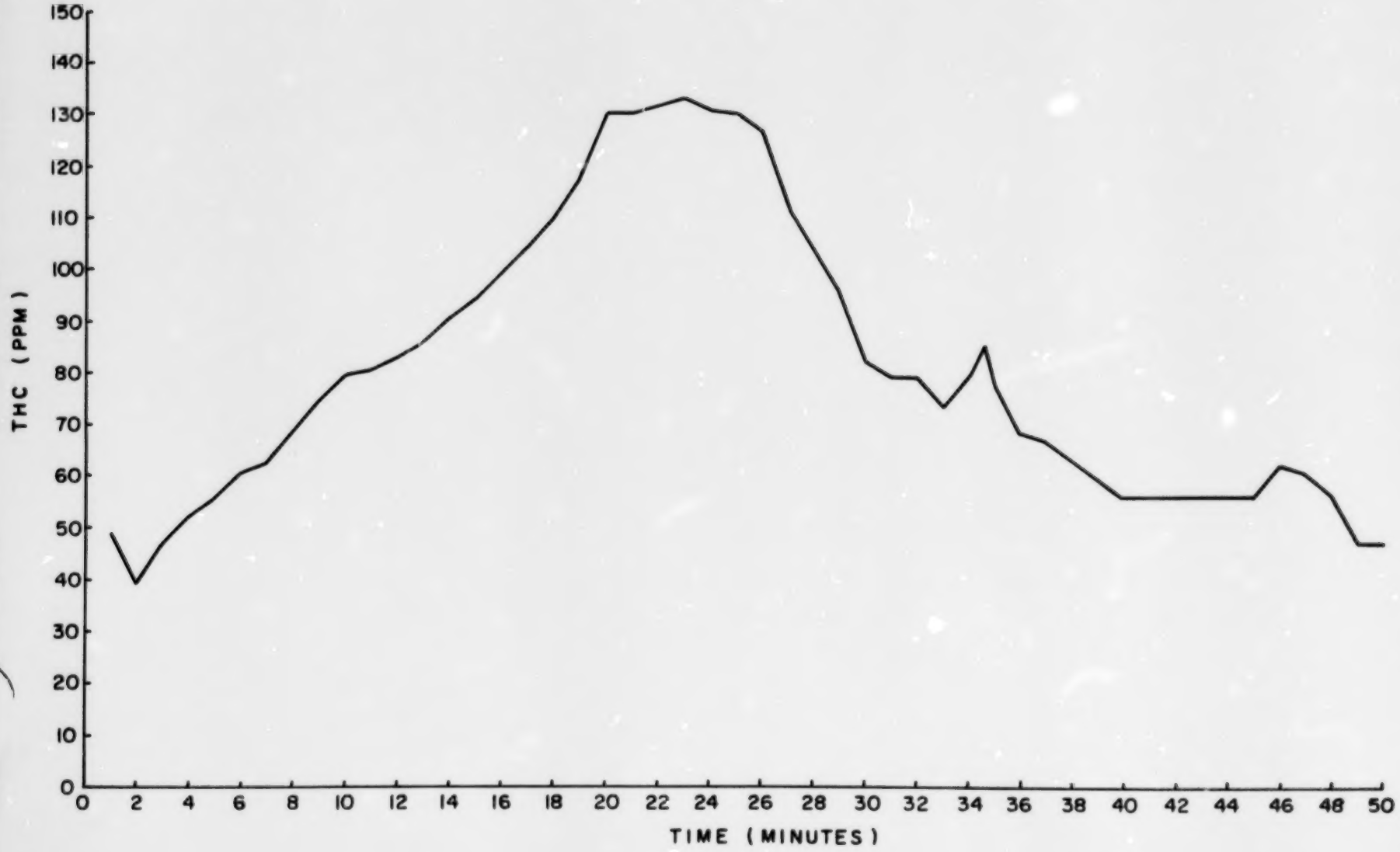


FIGURE 48 TOTAL HYDROCARBON CONCENTRATION vs TIME - BURNOFF No. 4.

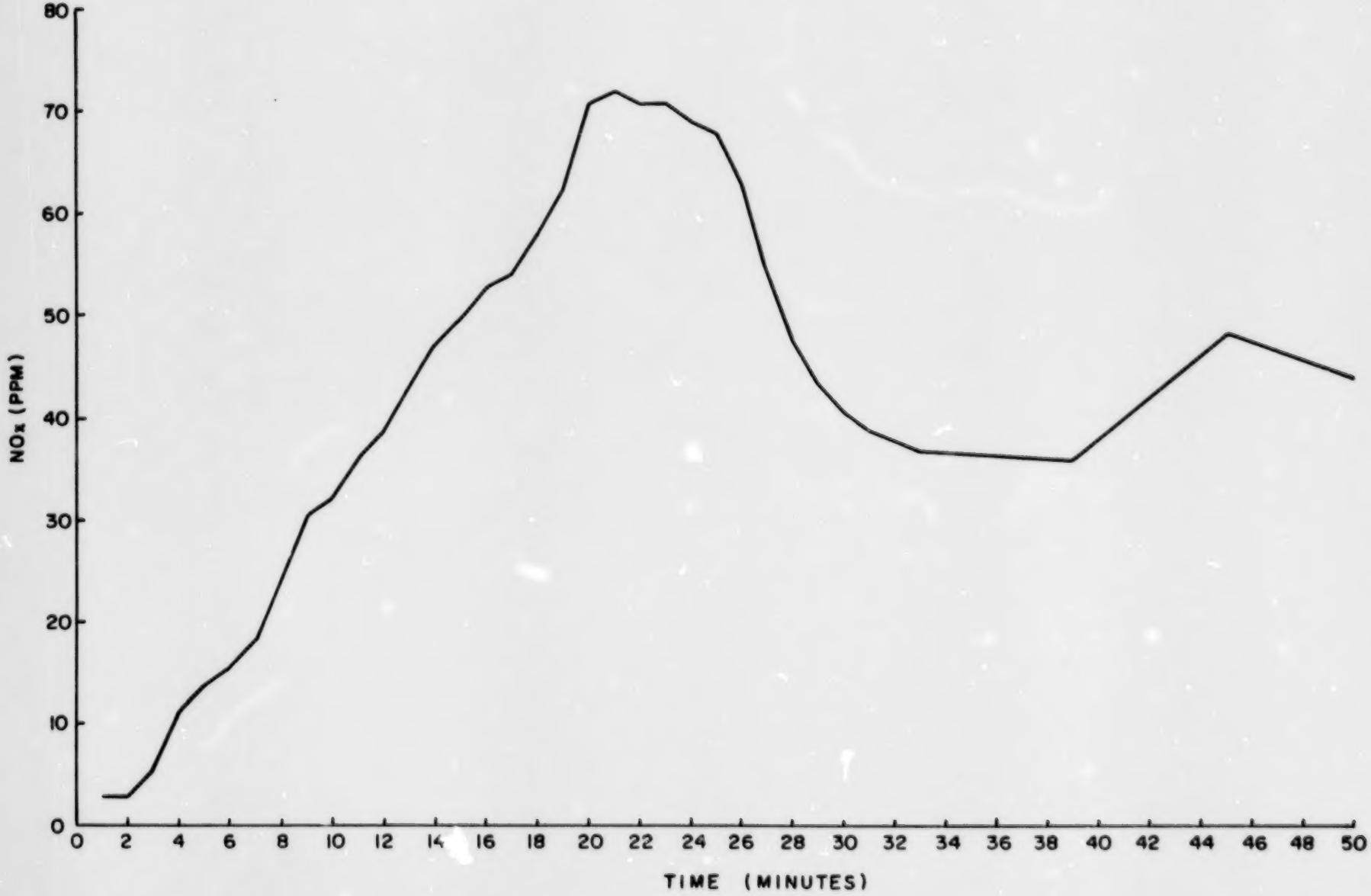


FIGURE 49 OXIDES OF NITROGEN CONCENTRATION vs TIME - BURNOFF No. 4.

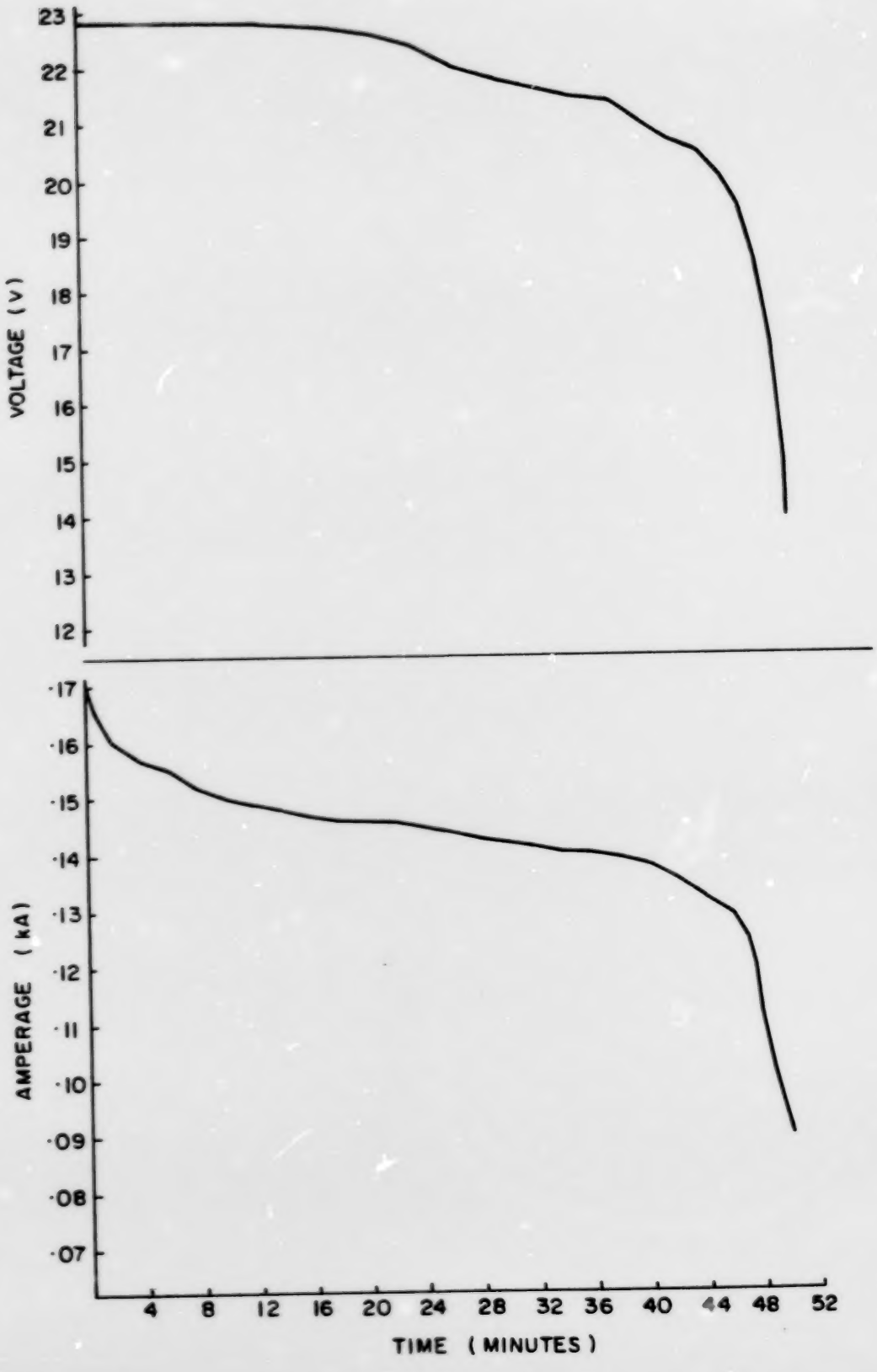
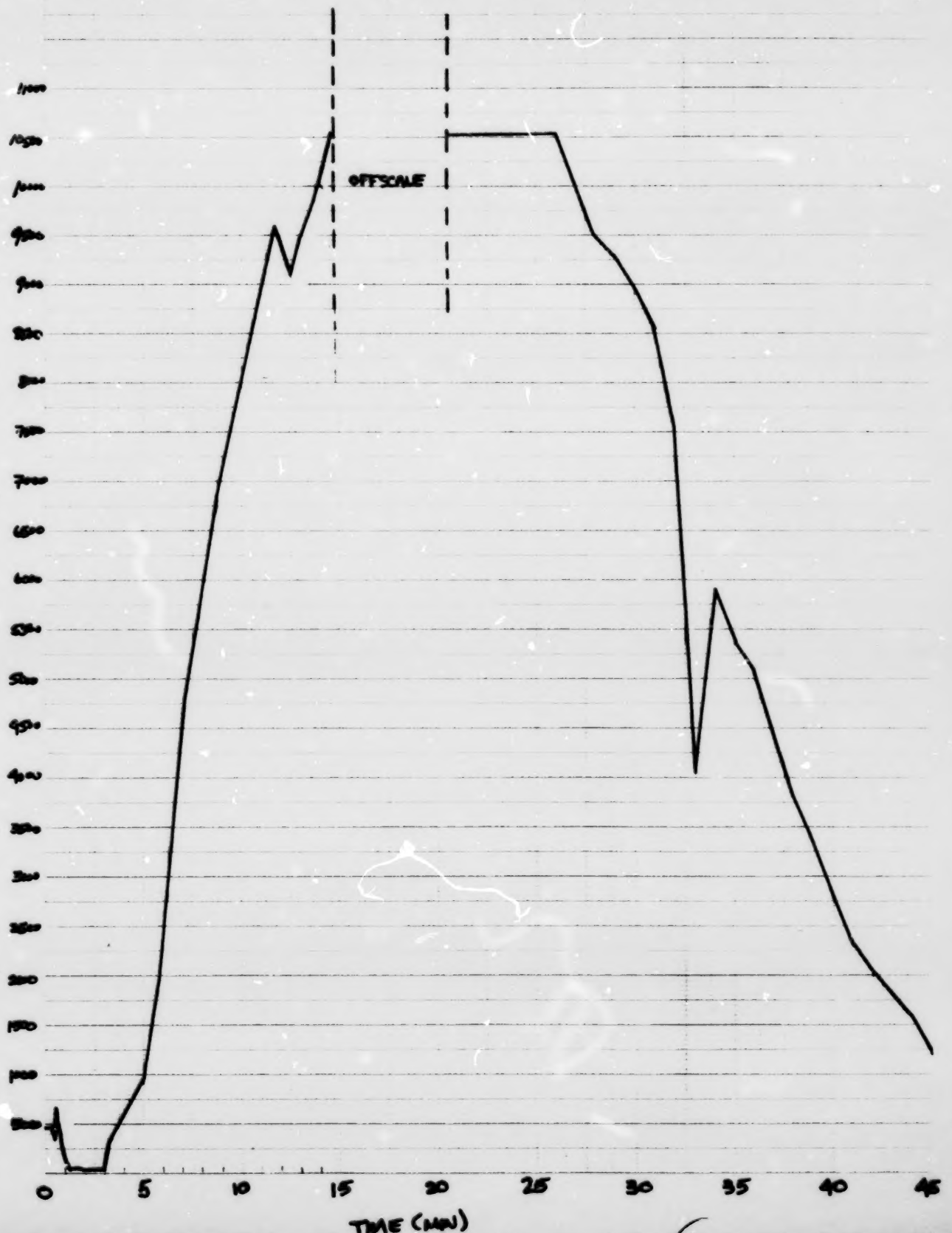


FIGURE 50 VOLTAGE AND CURRENT vs TIME - BURNOFF No.1.

APPENDIX

Gaseous Emission Concentration Profiles
For Burn Nos. 2 and 3

Figure 1: CO(PPM) VS TIME (MIN) BURN #2



46 1510

Figure 2: Corning Filter Burnoff NO, NOx Test 2

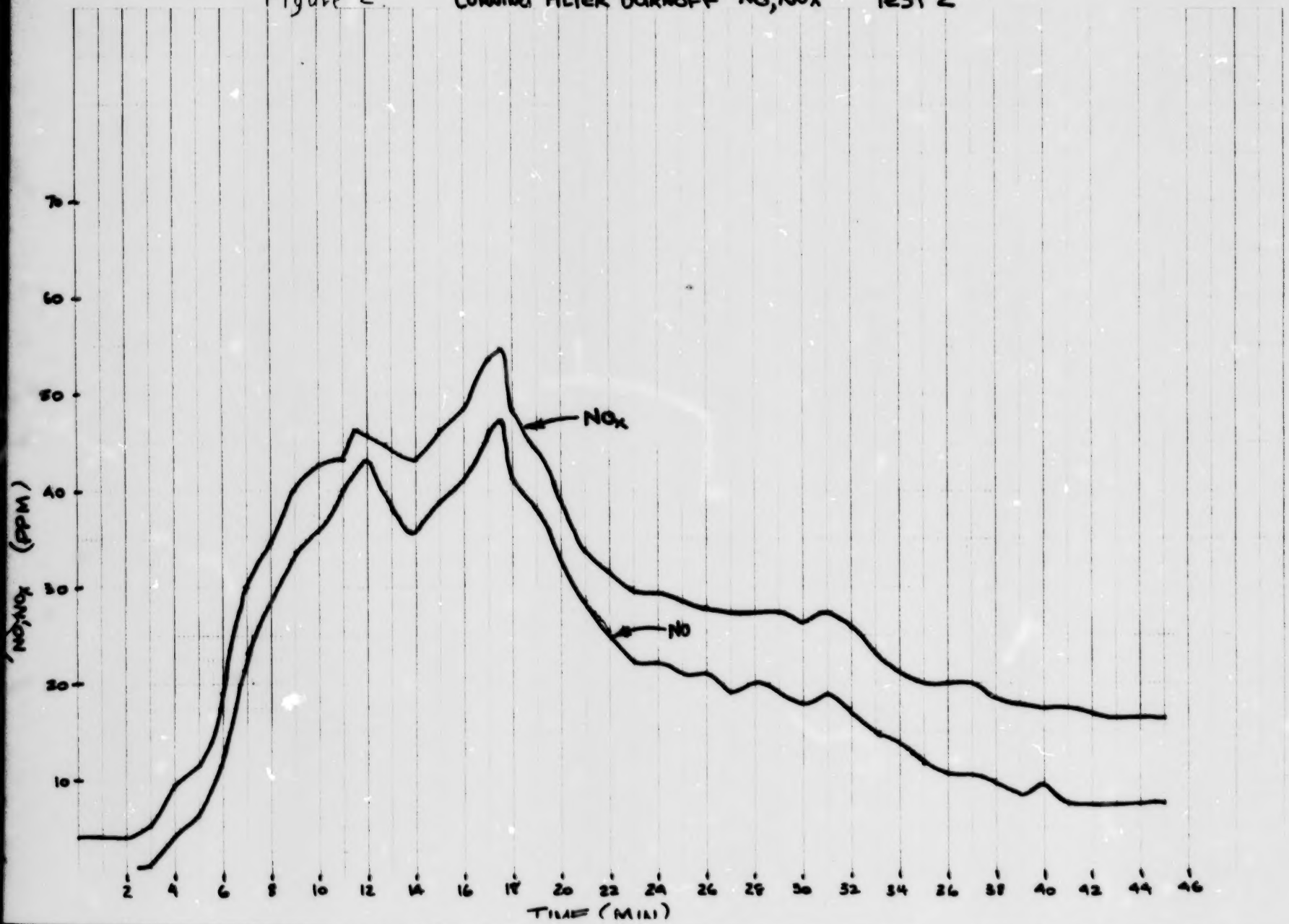


Figure 3: CORNING FILTER BURNOFF (CO_2) T.2. VS TIME (MIN.)

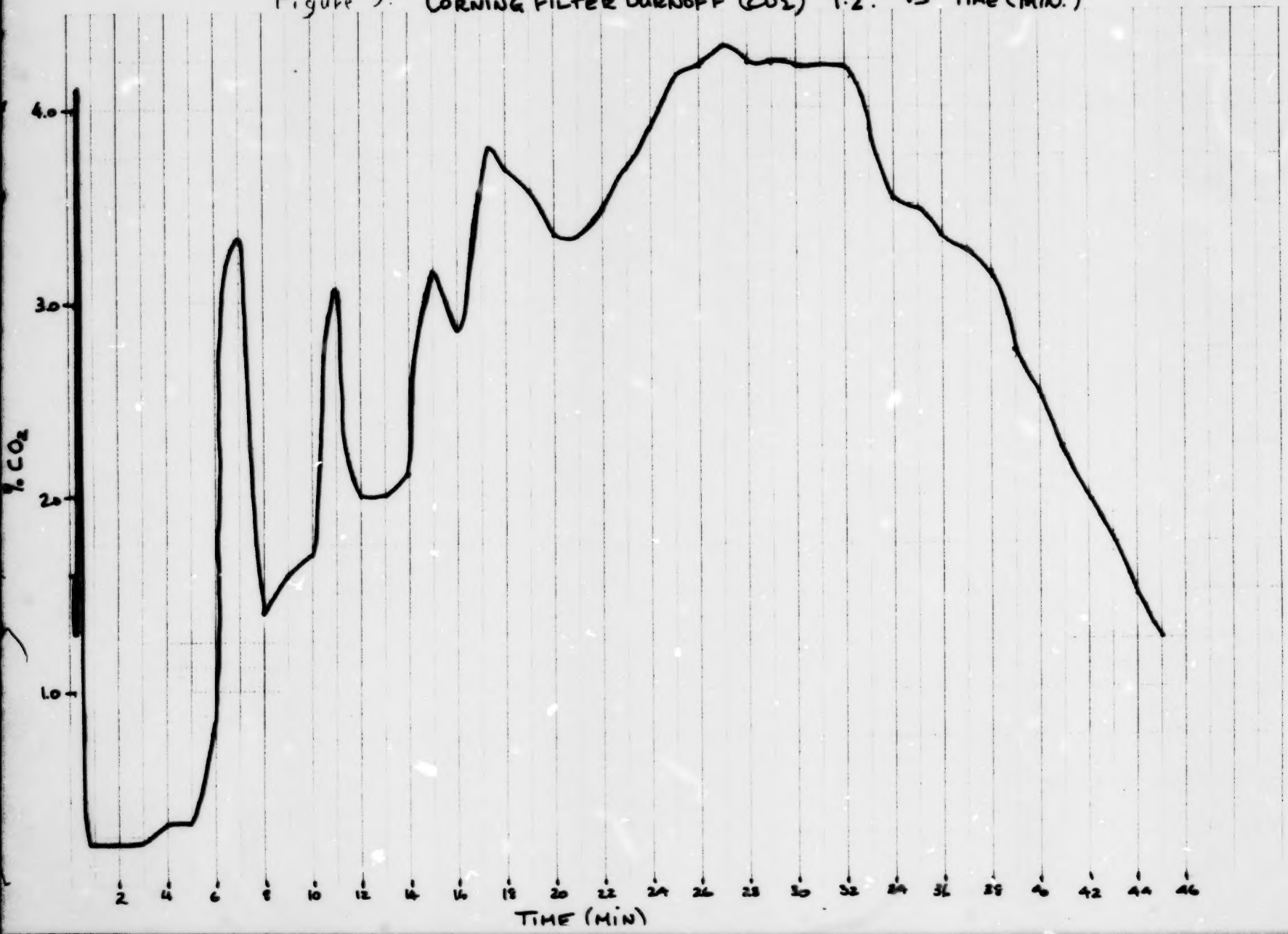


Figure 4: THC (PPM) VS TIME (MIN) BURN #2

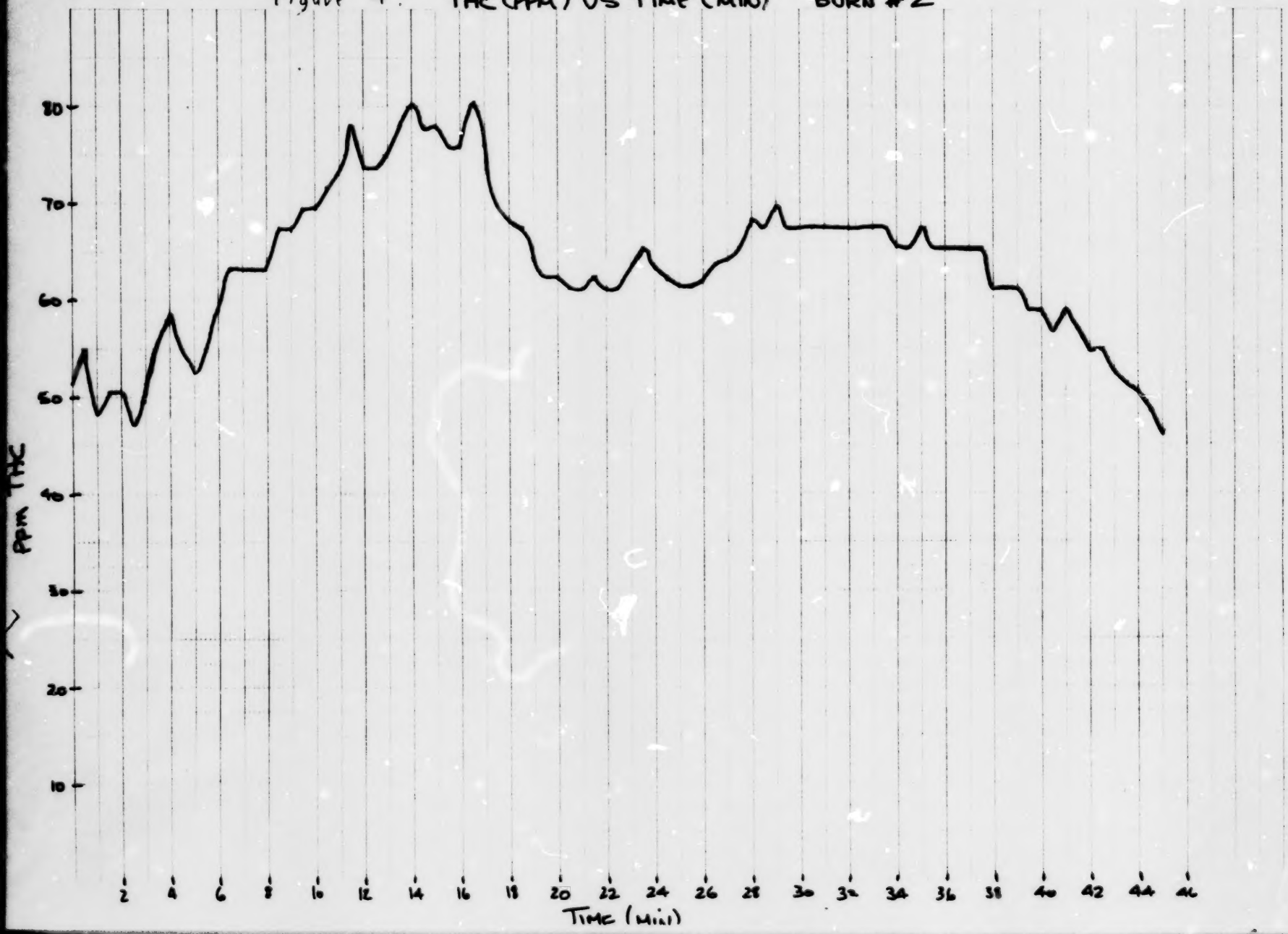
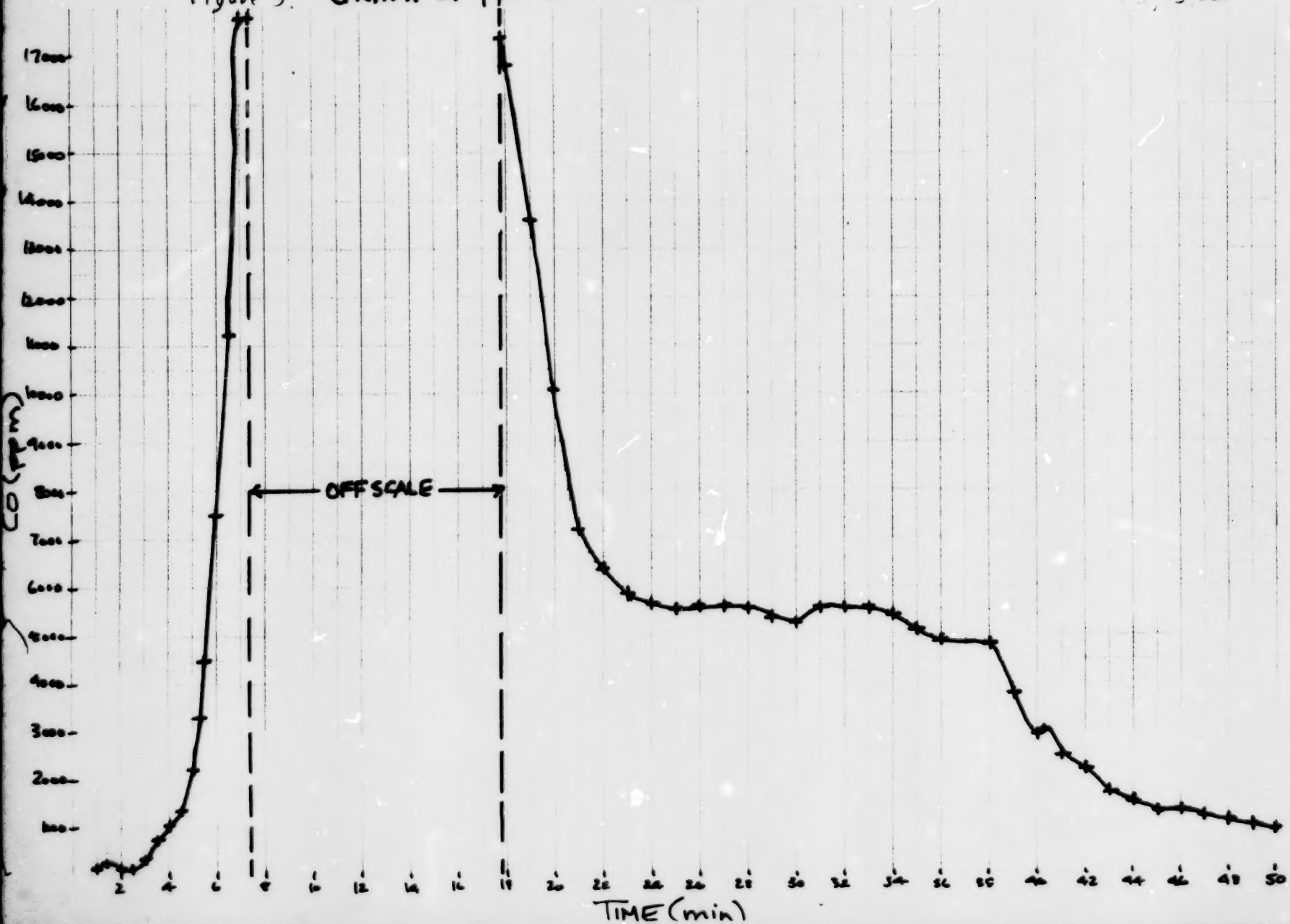


Figure 5: GRAPH OF ppm CO vs. TIME FOR BURN #3



461510

Figure 6: GRAPH OF %CO_2 VS. TIME FOR BURN #3

MAY 15/74

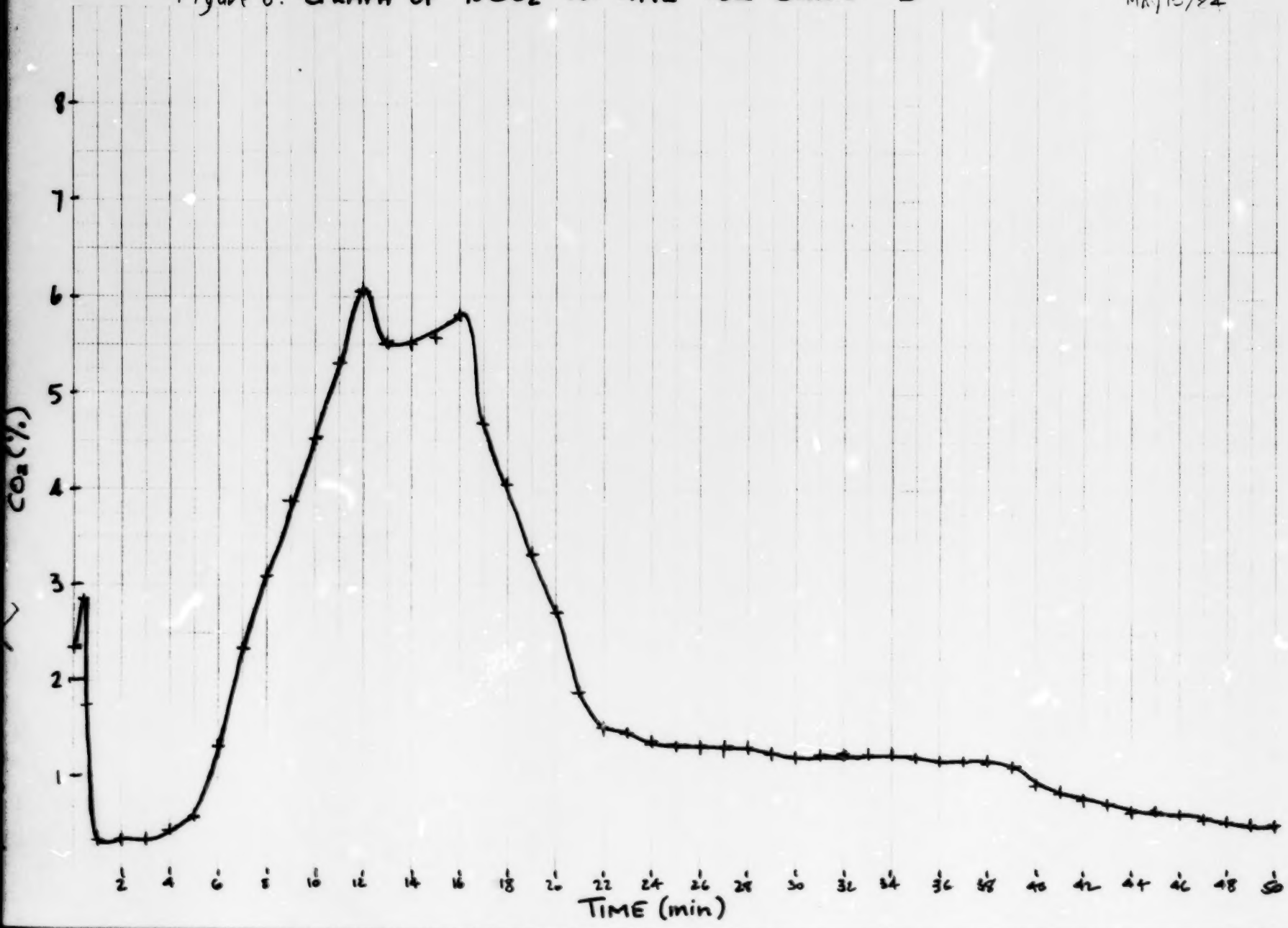


Figure 7: GRAPH OF ppm THC vs. TIME FOR BURN #3

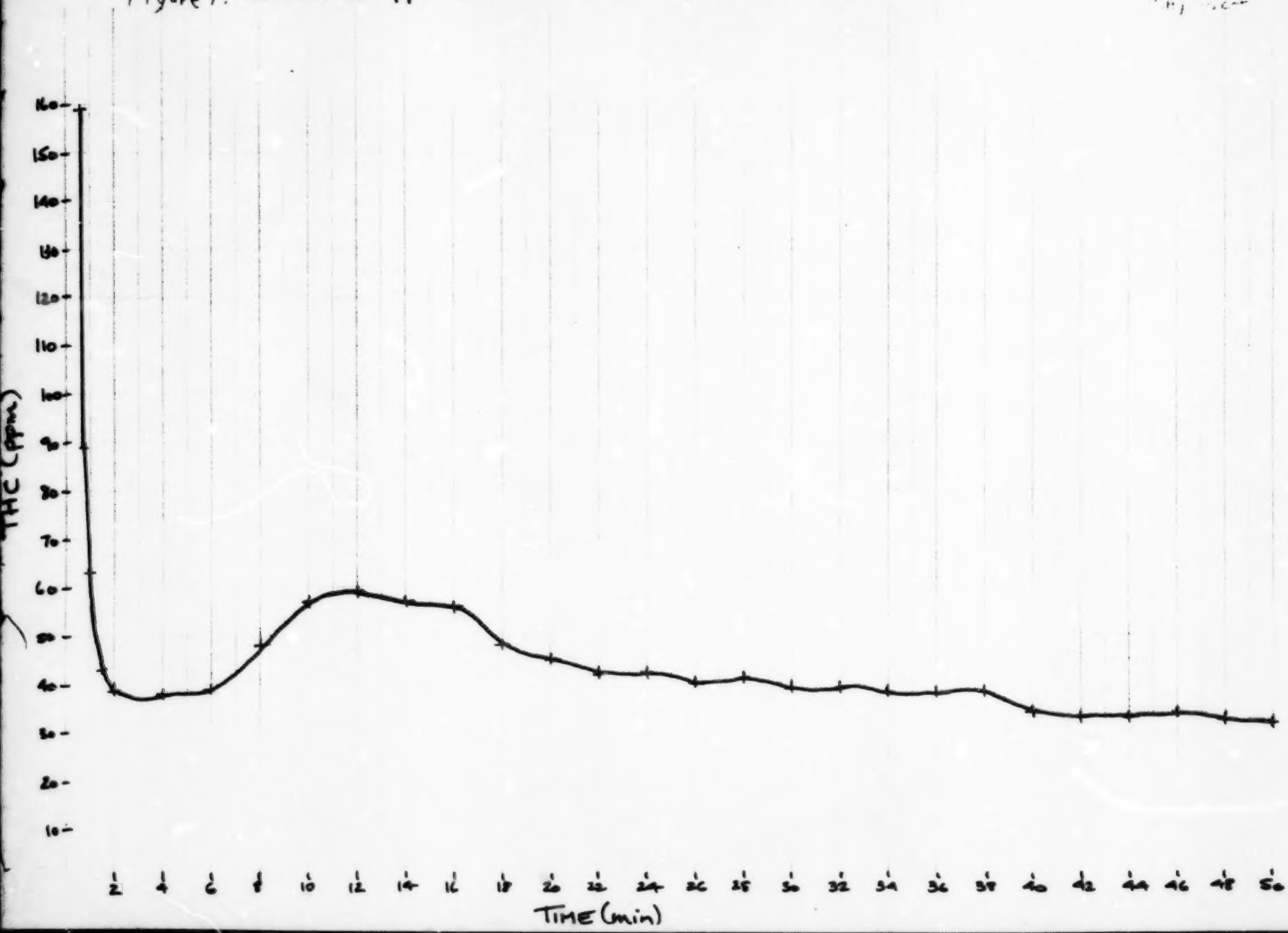
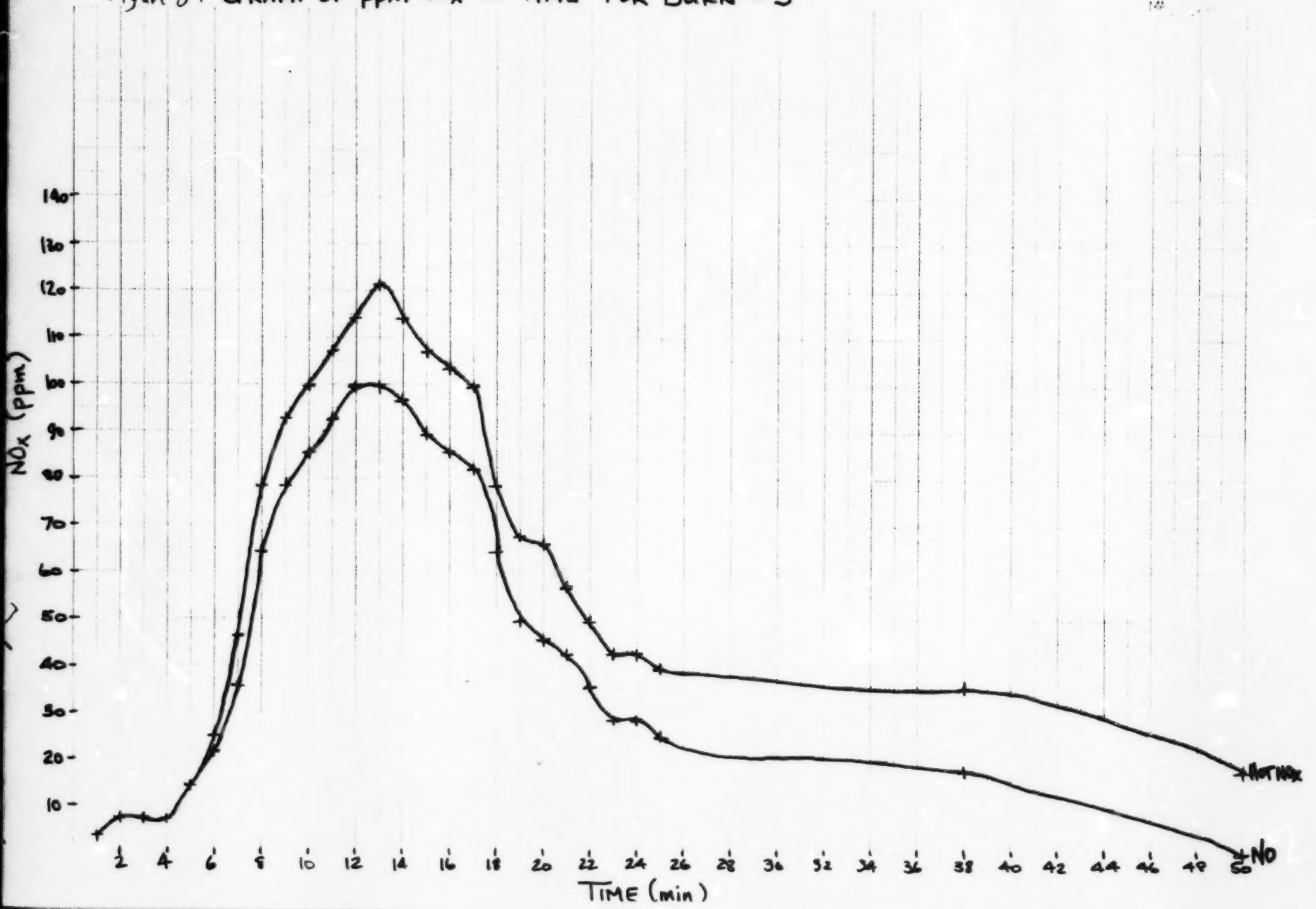


Figure 8: GRAPH OF ppm NO_x VS. TIME FOR BURN #3



ONTARIO**RESEARCH**
FOUNDATION

Energy

energy conservation programs and analyses, thermal insulation design and evaluation, waste heat recovery, building energy systems and monitoring, heat storage systems and materials, thermoelectric generation, combustion technology, burner development, heat transfer analysis, solar systems monitoring, National Solar Test Facility, solar technology, wind power, coal, lignite and oil sands processing, alternate fuel utilization, battery and fuel cell technology, peat processing and utilization, fenestration measurements, heat pump development and test facilities, residential heating systems, air to air heat exchange test facility, photovoltaic devices, . . .

Environment

ambient air quality assessment, environmental impact assessments, long-range transport of atmospheric pollutants, precipitation chemistry, particulate characterization studies, emission inventories, source sampling, odour emission and control, asbestos measurement and control, occupational health and safety studies, diesel emission control technology, air pollution control, pesticide residues, trace metal analysis, trace organic analysis, radionuclide analysis, mutagenic testing, noise and vibration analysis, radiation measurements, particulate identification and measurement, water surveys, water purification, industrial and municipal wastewater treatment, membrane separation techniques, wet oxidation, solid waste treatment and recovery, hazardous waste treatment and disposal, indoor air quality assessments, priority pollutant analysis, landfill technology, . . .

Materials

building materials, plastics, composites, metals, glass and ceramics, wood and wood composites, biomaterials, adhesives and printing inks, fibres, yarns, fabrics and geotextiles, leather, organic and inorganic chemicals, specification development, quality assurance, mechanical, chemical and thermal properties, test method development, failure analysis, fire and flammability, glass-metal seals, metal and alloy powders, metallography, materials characterization, materials characterization by optical and electron microscopy and X-ray microanalysis, photovoltaic materials, materials corrosion resource centre, corrosion properties and control, bacterial corrosion, coatings and sealants, rubber technology, X-ray analysis, cement and concrete, soils and aggregates, gypsum, body armour testing, footwear, paper-making materials, roofing and waterproofing, waste material stabilization and solidification, . . .

Products & Processes

clothing, footwear and industrial textile products, knitting technology, thin and thick film systems, microelectronics development, fibre optics, instrumentation, bioengineering, ultrasonic and fluid shear devices, microwave drying, transportation and equipment testing, mathematical stress and vibration analysis, product, equipment and machinery design & development, fuel emulsification, organic analysis and characterization, chemistry of foodstuffs, pulp and paper processes, ceramics processing, metallurgical process development, powder metal technology, packaging applications, technical and economic evaluations, industrial engineering applications, process control, enzyme analysis, electrical testing, radioactive surveys, industrial microbiology, biotechnology, technical and management skills development seminars, paper products, absorbent consumer products, detergent and cosmetic formulation, metal chemical analysis, flash hydropyrolysis of coal, electroplating, . . .

Resources

mineral processing, hydro and pyrometallurgical processing of ferrous and non-ferrous ores, coal evaluation, coal ash utilization and product development, asbestos processing and applications, non-metallic minerals/industrial minerals processing and utilization, uranium processing, forest utilization, utilization of forest and agricultural wastes, utilization of industrial, mining and domestic wastes, energy and chemicals from biomass, in-plant waste reduction, mineralogical services, mining and metallurgical waste disposal, . . .

ONTARIO RESEARCH FOUNDATION • SHERIDAN PARK, MISSISSAUGA, ONTARIO, CANADA L5K 1B3 • (416) 822-4111 • TELEX 06-982311

POOR

QUALITY

ORIGINAL